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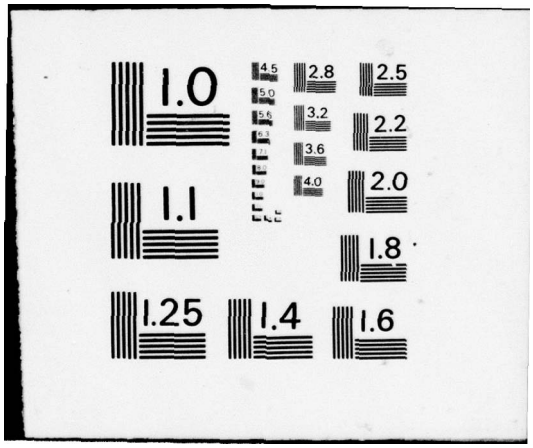
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REPORT SAMSO TR 76-92, VOLUME I

SECOND SURFACE THERMAL CONTROL MIRRORS FOR REFLECTION CONTROL

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GENERAL DYNAMICS CONVAIR DIVISION
SAN DIEGO, CA 92138

JANUARY 1977

FINAL TECHNICAL REPORT, VOLUME I

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This final report documents the results of a theoretical and experimental program to investigate ways to make second surface mirrors (e.g., thermal control surfaces, composed of thin transparent materials such as fused silica and FEP Teflon with a reflective backing, which are used on space vehicles) which are diffusely reflective but which retain the high solar reflectance of commercial specularly reflecting second surface mirrors. A number of designs were surveyed and four designs were fully evaluated. Three of these designs employed fused silica substrates with front or front and back surfaces ground with grinding

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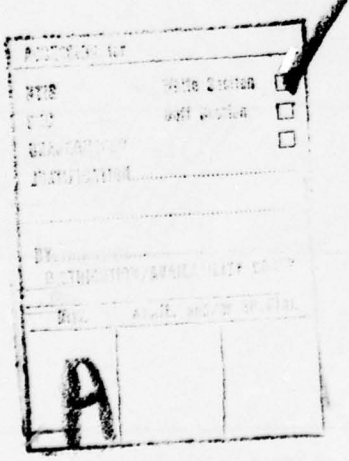
compounds and then etched in a hydrogen fluoride solution. When suitably silvered on the back sides, these specimens met design goals. One of these designs employed a FEP Teflon substrate with front and back surfaces contoured by compression of Teflon sheet between quartz plates in a vacuum oven. When silvered on the back side, good diffuseness was obtained but solar reflectance was slightly degraded over the reflectance of commercial Teflon second surface mirrors.



This technical report has been reviewed and is approved for publication.

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SECOND SURFACE THERMAL CONTROL MIRRORS FOR REFLECTION CONTROL VOLUME I FINAL TECHNICAL REPORT

10 JANUARY 1977

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GENERAL DYNAMICS
CONVAIR DIVISION

ABSTRACT

Thin transparent materials such as fused silica and FEP Teflon are used with a reflective backing as thermal control surfaces for space vehicles. These surfaces are commonly referred to as second surface mirrors. In this work a theoretical and experimental program was pursued to reduce the maximum BRDF for these second surface control mirrors to 2 sr^{-1} while retaining the high solar reflectance (94%) of commercial specularly reflecting second surface mirrors. A large number of designs were surveyed and four designs were selected for full evaluation.

Three of these designs employed a fused silica substrate. To scatter the incident radiation, the front or front and back surfaces of the fused silica blanks were thoroughly ground with fine grit grinding compound and then etched in an HF solution. The grit size, time of etch, and contouring or absence of contouring of the back surface were varied in the three fused silica designs. Specimens were obtained which, when suitably silvered on the back side, essentially met the design goals. Also, for special applications, data was generated on designs which would allow tradeoffs between the desirable characteristics of high solar reflectance, diffuseness, and diffuseness as a function of solar incidence angle.

One design employed FEP Teflon as the substrate material. Contouring of the front and back surfaces was accomplished by compression of Teflon sheet between quartz plates in a vacuum oven. The quartz plates were ground and etched in essentially the same manner as the fused silica mirror blanks. When silvered on the back side, good diffuseness performance was obtained but solar reflectance was slightly degraded over the reflectance of commercial second surface mirror Teflon. With more development work the reflectance could be improved.

FOREWORD

This report covers work performed at the Sensor Technology Laboratory, General Dynamics Convair Division for the Space and Missile Systems Organization, U.S. Air Force, Los Angeles, California, with Capt. William Lewis as Project Officer.

Contributors to the Project at Convair were:

Dr. J. T. Neu, Principal Investigator
Mr. M. F. Dorian, Performance Prediction
Dr. S. Kaye, Surface Contouring
Dr. F. Lopez-Lopez, Surface Characterization
Mr. R. S. Dummer, Optical Property Determination
Mr. W. T. Breckenridge, Computer Programming

Contributors to the Project at General Dynamics Pomona were:

Mr. Richard J. Sneed
Dr. Esther Krikorian

Members of SAMSO and the Aerospace Corporation through suggestions and recommendations contributed significantly to this development program. These contributors are as follows:

Capt. William Lewis, SAMSO
Dr. Charles Francis
Dr. Lester Goldstein
Mr. Robert Champetier
Mr. E. N. Borson

This work was performed under Air Force Contract F04701-74-C-0318.

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SECTION 1

INTRODUCTION AND SUMMARY

Spacecraft thermal control surfaces consisting of a thin transparent layer with a reflective back surface are currently available commercially in both rigid and flexible forms. Both are essentially specular reflectors. They are commonly referred to as second surface mirrors, however, their function is thermal control rather than production of a likeness. Indeed, the likeness producing quality of these surfaces in some cases becomes a burden. This work was undertaken to eliminate the "likeness producing" quality of these surfaces while retaining their thermal control properties. Simply stated, the objective of this program was the development of a surface configuration for both types of mirrors which would diffusely reflect light.

Initially, for the rigid mirrors, use of three substrates of fused silica (Corning 7940), micro sheet (Corning) and an adaptation of a diffuse coating already developed by Convair were considered. Very early in the program, it became evident that suitable methods did exist for contouring fused silica, and because of the superior space stability properties of this material, experimentation with microsheet was discontinued. SAMSO decided that development of a diffuse FEP Teflon* flexible coating was to be preferred to the development of the rigid Convair coating. The primary objective of this program, therefore, became the development of diffuse second surface mirrors using Corning 7940 fused silica as the substrate. The second objective was development of a diffuse mirror using flexible FEP Teflon as the substrate.

The program tasks were conveniently divided into two parts: substrate design and coating design. These two parts were pursued concurrently, based on the assumption that the quality (reflectance and adherence) of a coating for a diffuse mirror could be predicted based on the performance of the coating on a plain, smooth substrate. The validity of this assumption was verified in the course of this program.

The rigid second surface mirrors now available commercially are usually made of Corning 7940 fused silica. These are typically one-inch square, about 0.008 to 0.010-in. thick and are silvered on the back side. The primary design goal of this project was to equal the properties of the rigid specular commercial mirrors and in addition, reduce the bidirectional reflectance to $\leq 2\ sr^{-1}$. Selection of materials for stability in the space environment was required, but proof of stability was not given as a specific requirement of this effort.

The design goals and performance achieved for the rigid mirrors are shown in the following table:

*Trademark, E. I. DuPont

	Solar Directional Reflectance	Total Hemispherical Emittance	Bidirectional Reflectance
Design Goals (Commercial Specular Mirror Performance)	≥ 0.94	≥ 0.77	$\geq 2\ sr^{-1}$
Diffuse Sample Performance Achieved	0.942	0.775	$2.2\ sr^{-1}$

The design goals for the fused silica mirrors were essentially met and the cost of production runs of these diffuse thermal control surfaces should not exceed the cost of the standard mirrors.

Effective methods were developed to contour the Teflon to provide good scattering properties from a flexible material but the manufacturing process degraded the solar reflectance properties below the level required for good thermal control. However, where only moderate thermal problems exist, this diffuse, flexible material offers significant advantages over fused silica mirrors in initial cost, ease of application, and loss due to breakage. With some additional development work a practical flexible FEP Teflon material can be made which is a good solar reflector as well as a good diffuser.

In this volume (Volume I) the design and fabrication methods and rapid test methods are briefly discussed in Section 2, with detailed design theory and experimental methods detailed in annexes. Also in this volume are presented reflective coating design and application (Section 3); flexible substrate designs (Section 4); selection of designs for full scale testing (Section 5); and detailed manufacturing procedures (Section 6). Characterization of surfaces, a summary of surface optical properties and physical property testing are given, respectively, in Sections 7, 8, and 9.

Volume II contains the annexes which detail experimental optical measurements, test plan, and detailed optical data obtained on the developed mirrors.

Recommendations for further work based on the results of this program are as follows:

1. Pilot Production of Diffuse Fused Silica "Mirrors." Fabricate 100 of the fused silica mirrors developed by this program to verify production costs.
2. Flexible Materials. Complete the development of techniques for fabricating flexible FEP Teflon to provide a low-cost counterpart to the current flexible Teflon material. Methods of producing material in suitable sizes at acceptable prices would be part of the development. The ideas and, possibly, the techniques for

roughening the surface of Teflon could be applied to other flexible second surface thermal control materials.

3. Diffuse Solar Cells. Developing a solar cell which diffusely scatters reflected light but which is not degraded in electrical efficiency.

4. Laser Hardening. The laser hardness of the developed mirrors should be determined and possibilities for further hardening investigated.

5. Space Environment Testing. The diffuse fused silica mirror should be tested in simulated space environments to assure that the outstanding space stability qualities of the specular mirror have been retained (the likelihood of this retention is good).

SECTION 2

METHODS OF FORMING FUSED SILICA SUBSTRATES

2.1 SUBSTRATE DESIGN

The approach to designing the substrates for this program involved a combination of theoretical prediction and the use of experimental tests which could be rapidly performed. Emphasis was placed on the experimental approach.

It was concluded in early considerations of the task that the dual requirement of high reflectance and diffuseness, required that the substrate surface (front) or surfaces (front and back) be molded or shaped but also be microscopically smooth to refract the incident light without undue light absorptance in the substrate. A surface, as typically obtained from optical grinding, would cause multiple reflections of a beam in the substrate prior to emergence of the beam. The "scattering" performance would be good but the multiple reflections in the substrate would cause losses in reflectance and thus possible unacceptable degradation of the thermal control capabilities of the mirrors. A smoothly contoured surface would minimize the number of reflections prior to emergence of the beam and thus minimize reflectance degradation. The main thrust of the program was to determine the type of shaping or contouring required and how it could be obtained. Theory was useful in determining the type of contouring which would be useful.

The theoretical approach is presented in detail in Annex X.

2.2 METHODS

Five primary and two secondary contouring methods were tried for producing suitably shaped fused silica substrates. The primary methods roughened the surfaces; the secondary methods, applied to the roughened surface, smoothed out the roughness on a microscopic scale. Application of the primary and then the secondary method was required to obtain substrates in the acceptable optical performance range.

Primary Methods

1. Scribing
2. Laser beam contouring
3. Grit blasting, sand blasting and vapor and liquid honing
4. Ultrasonic impact grinding
5. Abrasive grinding

Secondary Methods

1. Fire polishing
2. HF etching

After considerable experimentation with these methods, the superiority of ultrasonic impact grinding and abrasive grinding followed by HF etching became apparent and efforts to develop suitable substrates were concentrated on application of these procedures.

The secondary procedures are discussed first and in general terms, followed by discussions of the primary procedures with comments where applicable regarding the secondary procedures.

In order to allow testing of a wide variety of concepts, the usual procedures for the determination of bidirectional reflectance and solar absorptance were modified so that a candidate could be optically tested in a few minutes and the essentials of performance determined. Selection of the four designs for the full-scale testing was based on the quick look data. To perform the quick look tests, it was necessary to apply a "utility" silver coating. This utility coating was made by a rapid evaporation of about 1800 Å of silver onto the substrate. The substrates were cleaned by vapor degreasing. No adhesion layer of aluminum or aluminum oxide was applied. Further, no protective coating was applied over the silver. The directional-hemispherical and bidirectional reflectances were determined as described below.

2.2.1 DIRECTIONAL-HEMISPHERICAL REFLECTANCE. For the directional-hemispherical reflectance "quick look" measurements, use was made of the Cary Model 14 with integrating sphere as described in Annex I of Volume II. Measurements were made at fixed wave lengths of 0.4, 0.45, 0.5, and 0.55 μm; the full spectrum was not scanned. At the same time, an uncontoured silvered blank was measured.

2.2.2 BIDIRECTIONAL REFLECTANCE. For "quick look" bidirectional reflectance, use was made of the bidirectional reflectometer described in Annex III of Volume II.

A reference method was employed. For given angles of incidence, θ, and reflectance, θ', the light ($\lambda = 0.45\,\mu m$) reflected from a standard sample was measured; the standard was then replaced with the sample second surface mirror and the reflected light again measured.

2.3 FIRE POLISHING

A surface roughened by grinding, grit blasting, vapor honing can be smoothed by fire polishing with a gas-oxygen flame. Application of the flame to 0.010-in.-thick wafers is impractical; the heat distorts the flatness of the wafer and makes it curl at the edges.

In laboratory experiments a 0.040-in.-thick wafer of fused silica was lapped with varying grit sizes and a gas oxygen flame played on the surface. It was planned to reduce the waver thickness to 0.010 in. after flame polishing if the polishing was successful. It was found that fusion of the lapped surface could be obtained and a transparent, microscopically smooth, "bright" wafer surface obtained, but alas, the surface was too smooth; its reflected light was insufficiently diffused. Further, the process was difficult to control. Variations in the degree of fusion from sample to sample and from area to area on the face of the same sample could not be eliminated. Further experimentation was dropped.

2.4 HF ETCHING

Liquid phase HF etching was found to be highly effective in transforming abrasion roughened surfaces into microscopically smooth surfaces. Vapor phase etching was not as effective. Both types of etching are discussed below.

2.4.1 LIQUID PHASE ETCHING. Freshly abraded fused silica surfaces appear similar to paper under high magnification (see the top four pictures in Figure 7-1 The outer surface consists of distributed glass debris over a field of microcracks in the fused silica surface. The debris is sufficiently strongly held onto the substrate that it is not removed by working or rubbing. As noted in Section 2, these surfaces are good diffusers. But, by their nature, they refract an incident ray many times before it is reflected from the SiO_2 silver interface and is ultimately scattered. Since each refraction involves a small absorption of the ray, this type of surface cannot provide the ultimate in high reflectance.

On exposure to an HF solution, the loose particles which have been pressed and fused at corners and edges, gradually disappear revealing deeper grooves and cracks in the glass. Continued HF attack dissolves away all loose material and causes sharp edges to disappear and grooves to widen and intersect. Scanning electron microscope photographs at this stage show the surface to have a worming appearance. On further etching, the grooves, cracks and depressions form into intersecting smooth depressions. Continued etching results in larger and shallower depressions.

The process is illustrated in Figure 2-1, which shows an unetched abraded area on the right, a 1.5-hour etched surface on the left, and a continuous transition region in between. The worms in the transition region appear to result from the acid "cleaning out" the microcracks in the fused silica.

Changing grit size changes the scale but not the general process. This is illustrated in Figures 7-1 and 7-2. Figure 7-1 shows scanning electron microscope pictures of an array of fused silica samples contoured using different grit sizes and times of HF etching. All photographs were taken at the same magnification, 300X. Thus, a segment of 15 mm is equivalent to 50 μm on the surface. The photographs shown in Figure 7-1 are taken at an angle of about 30° to the normal to the surface, and represent

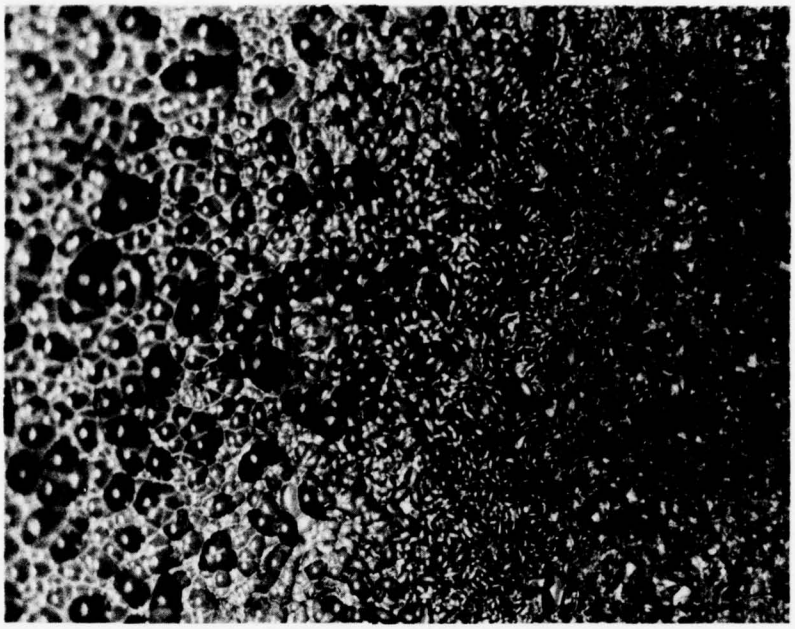


Figure 2-1. Transition Region between an Area Etched for 1.5 hrs (at Left) to an Unetched Region (Right)

a top view of the surfaces. The photographs are arranged in order of increasing grit size from left to right (3, 9, 22, and 177 μm) and increasing time of etching from top to bottom (0, 0.5, 1, 2, 2.5 hours).

It may be noted from these photographs that the finer grits and longer times of etching seem to give a greater degree of randomness. Also, there is a correlation between the average area of these depressions and the size of the grit used to produce them. It may also be observed that the desired surfaces of overlapped conchoidal shapes are achieved with substantially less etching time for small grits than for large ones. The surfaces are "microscopically smooth" which is the type of surface required for this development. Experimental measurements show that reflectance is increased by time of etching and use of small grit sizes; diffuseness, on the contrary, is increased by short etch times and coarser grit.

The profiles of the surfaces photographed with the scanning electron microscope (Figure 7-1) have also been photographed directly with an optical microscope and a Polaroid camera. The results are given in Figure 7-2, arranged in the same manner as Figure 7-1 for comparison. The same magnification was used (X300).

The samples were bonded together (see Figure 7-4) with pitch and the edges were ground and polished with successively smaller grits down to 3-μm size grit.

As noted in the figure, it was not possible to achieve a smooth enough polish to produce well cut, sharp edges. Because the pitch that holds the samples together is softer than the silica, and because the heat generated in the process of grinding and polishing, the material tends to crack and chip precisely at the edges to be photographed. Despite the presence of some chips, the general shape of the profiles may be clearly appreciated from the photographs.

In the liquid phase HF etching of SiO_2, the parameters investigated were: (1) temperature, (2) HF concentration, (3) time of etch, (4) agitation during HF etching, and (5) substrate cleanliness.

2.4.1.1 Temperature. Elevated and depressed temperature etching was performed. Elevated temperatures naturally accelerated the rate of reaction and depressed temperatures slowed the rate. No difference in the quality of the resultant surface was found, irrespective of the solution temperature; therefore, a conveniently reproducible temperature of 23° ±2°C was adopted for all etching.

2.4.1.2 HF Concentration. An HF concentration was selected to provide adequate speed of etching while producing a smooth uniform pattern. Concentrated acid is harsher and does not produce as uniform a surface as the slightly diluted (2:1 HF to water) mixture. The concentrated acid is more apt to develop pits and attack the softer or more alkaline zones (this was quite evident with soft glass substrates). Very diluted acid on the other hand, did not produce any better results than that obtained with the 2:1 mixture and, therefore, the 2:1 HF:H_2O acid concentration was selected.

2.4.1.3 Time of Etch. A certain minimum time for each coarseness of grinding was required to remove all loose debris and coalesce the depressions. Etching past this clean-up time resulted in variations in optical performance; best light scattering was obtained with minimum etch times, but solar reflectance improves with time of etch. These effects are discussed in detail in Section 5. The etching process removes between 0.001 in. and 0.0013 in. of material per hour per side of the sample, when immersed in the solution of HF.

2.4.1.4 Agitation During HF Etching. All chemical reactions may be expected to proceed more smoothly and uniformly when the system is agitated. Old or used reactant and products are removed from the reaction zone and the renewed surface exposed to constantly flowing fresh reactant. In etching the fused silica substrates, however, it was found that static exposure in individual disposable polyethylene beakers gave perfectly satisfactory results; therefore, agitation was not used for production samples.

2.4.1.5 Substrate Cleanliness. A factor which affects results of the substrate etching is the nature and extent of clearing. Occasionally, etched substrates were noted

to have a mottled appearance. This was attributed to some slight contamination on the abraded surface when etching was performed, but the presence of strains in the fused silica which selectively etched cannot be ruled out. The problem was more cosmetic than real. No optical performance variation from similar substrates with and without the mottled appearance was found.

2.4.2 VAPOR PHASE ETCHING. Vapor phase etching is more localized than liquid phase etching. This may be due to droplet formations on reaction at nuclei or active spots. In any event, vapor phase etching is a slower procedure and produces a frosted appearance, i.e., microscopically "rough" in contrast to microscopically "smooth," using solution. Clearly the latter was required in this program.

2.5 SCRIBING

A few experiments were done to attain a pattern by scribing. The scribing procedure was done very simply. A glass blank was affixed to a holder. A glass cutter or a diamond scribe used as a glass marking pencil was then used with a ruler to imprint a cross-hatch pattern on the surface. Some variation in the impressed pattern could be obtained by varying the pressure applied on the glass. However, it was difficult to achieve any "wavy" smooth patterns.

Scribing alone produced scratches and cracks in the fused silica and without application of secondary forming methods did not meet reflectance requirements. Fine polishing this type of substrate (and other types also) proved not to be satisfactorily reproducible and also the patterns formed were too regular (smooth) and did not meet the diffuseness requirements. HF etched scribed substrates were inferior to HF etched ground substrates and accordingly scribing was discontinued in favor of grinding.

2.6 LASER-BEAM CONTOURING

Early experiments with a CO_2 laser beam showed that the method could produce contouring of the fused silica surface without breaking the substrate.

The experimental set-up was as follows:

A 100-watt laser (Coherent Radiation Lab) was energized. The beam which was about 15 mm in diameter was focused to a point at the workpiece by means of a germanium lens with a focal length of 2.5 inches.

The arrangement of the components is shown in Figure 2-2. The laser was mounted on a Portelevator, an adjustable-height table. It was moved with its power supply, gas tanks and control unit to a Bridgeport milling machine. The workpiece which consisted of a microscope slide, Pyrex plate, or fused silica square was mounted on the table of the milling machine so that it could be accurately indexed for vertical motion

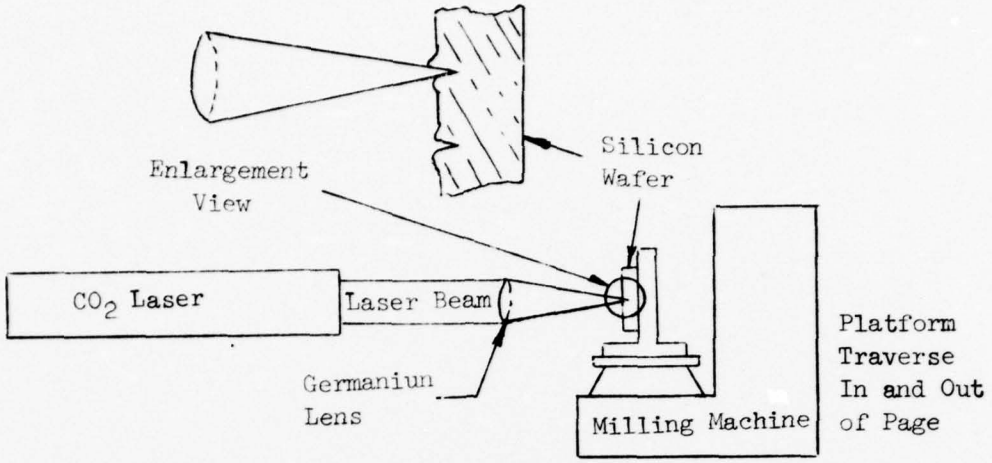


Figure 2-2. Arrangement of Laser Contouring Components

and closely regulated in speed for linear motion transverse to the beam. The laser controller permitted manual or automatic operation in a continuous or pulsed mode. In the pulsed mode, the pulse frequency and pulse width could be regulated.

Prior to a test on a fused silica specimen, a piece of heat-sensitive paper was placed in the focal plane of the lens. By manipulation of the milling machine table, the focal point was adjusted to graze or to penetrate the specimen to the depth desired. The specimen was then installed on the milling machine table and the laser beam adjusted to impinge on a corner of the specimen. Manual pulsing permitted one to select a mode of operation in which the pulse width provided the desired energy for contouring the fused silica surface. The frequency of repetition together with the linear travel rate at right angles to the beam produced the desired contouring.

A high pulse frequency and wide pulse period produced a "line" contour, especially with low rates of linear travel. A ridge pattern was obtained by proper indexing of the lines vertically. Careful adjustment of the focal point of the beam just at the specimen surface or below the surface determined the width of the "ridge" or the radius of the molten depression made by an intermittently energized beam of energy. The automatic modes of operation produced two types of regularities which were considered undesirable. First, was the system of lines or rows of contours one above the other. Second, was the regularity of pulsing which gave regularly spaced depressions along each line. The duration and frequency of an intermittent beam is readily controlled. Although the laser beam produced thermal shock patterns on soft glass surfaces, fused silica blanks accepted the impact satisfactorily so that each pulse produced a depression. The depth of penetration of each explosive laser discharge was easily varied by adjustment of the laser controls and the distance from the surface of the blank to the laser focusing lens. The surface configuration generated by this technique consisted

of a series of depressions with a raised periphery caused by rapid chilling of molten material. This outline closely resembled lunar craters.

Insufficient power on fused silica caused apparent deposition of condensed silica at the perimeter of the laser-produced hole. In the continuous mode, V-grooves were cut in which powdered silica was deposted. HF dissolved much of the powdered silica, but the deeper parts were not affected.

At this stage of development, the grinding technique was shown to produce good results faster. Also, the mechanics of laser forming produced regularities of pattern which were undesirable. Therefore, the method was not studied further.

2.7 GRIT BLASTING

Grit, ejected at high velocity by a stream of water or air, provides a rapid method of contouring fused silica. Several applications of this method were tried as follows:

Grit Size (μm)	Transport Agent
149	Water
149	Dry Air
36	Dry Air
27	Dry Air

On a macroscopic scale, the surface finishes appeared uniform; but on a microscopic scale, it was found that the method produced a large amount of erosion and relatively large deposits of debris. Scanning electron microscope (SEM) pictures showed grooving, cracking, spalling, pitting, and uneven accumulation of detritus in crevices and corners. On HF etching, the acid first dissolved the small, loose glass particles. Short periods of exposure, or dilute acid allowed some of this to remain as contamination attached to the surface. Apparently sufficient heat is generated locally during abrasion so that fusion occurs at asperities. Also, micro-cracks which arise in all abrasive methods were very prevalent. NBS Publication 348[1] contains many of the finer details and explanations of phenomena occurring during the grinding and etching process. After some of the debris was cleared by HF, zones appear at high points, which seem to consist of fractured or crushed glass which had not crumbled away. After more prolonged contact with acid, the small chips and debris as well as the broken material disappeared. The final surface then presents an appearance of closely meshed crescent shaped depressions with a rather narrow distribution of wall-to-wall diameters. Further action of the acid results in formation of larger diameter depressions as the walls of smaller crescents merge into fewer and wider shallow depressions.

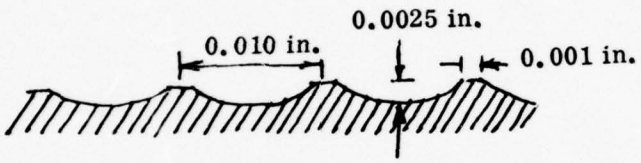
The ultimate contoured surfaces obtained by these methods were similar in form to the surfaces obtained by lapping and etching. The grit blasted surface in the experiments

performed were in general coarser than the lapped surfaces and showed poorer optical performance than the lapped surfaces and effort was therefore concentrated on the lapped surfaces. It is possible that the end product obtained by lapping could also be obtained by variations on the grit blasting methods. But, even if this were the case, the lapping method would be preferred for its simplicity and economy of application.

2.8 ULTRASONIC IMPACT GRINDING

Abrasive particles may be propelled onto a surface and thereby cause abrasion, with a tool vibrating at ultrasonic frequencies. An ultrasonic impact grinder, manufactured by Sheffield Corporation, was used in this work to shape silica wafer surface.

A spiral pattern was cut in the copper with a rounded tool. A cross-section of the spiral was as follows:



It was noted in viewing impacted samples under a microscope that the grooves tended to become perceptibly shallower after about six or seven samples were made with a new tool. As an operating procedure the first six samples made with a new tool were categorized "deep," the second six "shallow." The tool was refaced after manufacture of 12 samples.

Impact ground surfaces were etched after grinding — just as the flat ground surfaces were etched.

The impacted samples were made from 1-in.-square fused silica blanks, 0.040-in. thick, polished on one side. The first operation was to impact the polished side. Thick (0.040 in.) starting blanks were found to be a requirement. The thin blanks broke up when impacted. A polished surface made easy the determination of complete mating of the tool and the fused silica.

Substrates were finished to about 0.009 in. by conventional optical fabricating methods after impacting. Impacted samples which were to have a 3 μm side were pitched to a 6-in.-diameter iron tool, impacted side down and the non-impacted side was reduced in thickness with a generator, rough ground, fine ground, and finally "ground out" with 3 μm grit. Impacted samples which were to have a polished side were etched after impacting, then pitched down, reduced in thickness as noted above and paper polished.

2.9 ABRASIVE LAPPING

Good results were achieved by lapping fused silica wafers with different grits of various sizes and finishing with an etch of hydrofluoric acid (HF) for selected periods of time. This HF treatment removes the siliceous debris which was rather loosely fused on the surface, and smoothed the resulting undulating contours. The process was readily controlled.

Fused silica is a homogeneous material and etching occurs uniformly over the entire surface. It is a high temperature material which is very viscous in the glassy state so that hardly any nucleation or devitrification is observed after etching the ground surfaces. (It has a slow etching rate compared to glasses.)

The variables investigated experimentally were:

1. Grinding compounds
   a. Alumina, Al_2O_3
   b. Silicon Carbide, SiC
2. Particle sizes 3, 5, 7, 9, 15, 22, 30, 177, 250 μm
3. Surfaces
   a. Single side
   b. Both sides

2.9.1 GRIT MATERIAL. Silicon carbide, SiC, was used in the coarser grades, 22-250 μm. It cuts fast, has sharp edges and has a greater tendency than alumina to produce micro-cracks. Subtle differences were noted in the finish produced by Al_2O_3 and SiC before and after etching. Al_2O_3 was used for grinding at the finer sizes, 3 to 15 m. Al_2O_3 is comparatively soft and has breaking and cutting characteristics which produced a fine surface on fused silica.

2.9.2 PARTICLE SIZES. About 15 particle sizes were used in attempts to achieve the desired optical characteristics with the final product. Type and sizes were somewhat limited by availability and common usage in the optical shop.

2.9.3 SURFACES. Some fused silica substrates were contoured on both sides, and some were contoured on one side only and the other polished. In initial work when one side only was to be ground and etched, the opposite side was to remain polished, a coat of beeswax was brushed on the polished surface to protect it during etch. It was found that a better method was to grind a relatively thick (0.022 in.) blank on one side, etch the entire blank, then grind and polish the side that was initially "unground."

2.9.4 LAPPING. Lapping may be accomplished manually or with optical lapping machines.

2.9.4.1 Manual Lapping. The work piece is manipulated on a flat base by hand using wet grit as an abrasive. The method is convenient and fast. Results are fairly satisfactory. However, the thin (0.010 in.) square blanks are not easy to lap. Thin stock breaks very readily and also bends. Care and continuous observation is necessary to achieve a uniformly ground sample. Further, only one piece can be ground at a time. Machine grinding is generally preferred.

2.9.4.2 Machine Grinding. The preferred method of grinding and the one which gives the best results faster and with several pieces simultaneously is to use optical machine tools. In this method, several blank specimens are attached to a disc with hot beeswax. All must be of the same nominal thickness. Grinding then proceeds on an optical grinding machine with periodic application of a slurry holding the appropriate grit. When the desired finish has been achieved, the specimens are detached and cleaned in trichloroethane and acetone.

This method is fast and precise and is preferred for large-scale production.

SECTION 3

REFLECTIVE COATINGS

The General Dynamics Pomona Division designed the reflective coatings for the fused silica substrates, coated the contoured substrates which were used by General Dynamics Convair Division for testing and did certain developmental tests as required in developing the designs.

Two types of coatings were developed, both of which find counterparts in commercial specular second surface mirrors:

- Regular or non-enhanced silver coatings
- Enhanced silver coatings

However, the coatings developed by General Dynamics are not necessarily the same as the commercial mirror coatings (the exact nature of commercial coatings is not known).

Testing at Pomona included:

1. Specular reflectance measurements at near normal incidence (UV-IR).
2. Scotch tape adherence tests.
3. Boiling water and subsequent scotch tape tests.
4. Thermal cycling between specified limits.

Final testing efforts were carried out in San Diego at Convair.

The approaches which led to the successful preparation of both unenhanced and enhanced second surface mirrors were as follows:

1. The principal second surface mirror material was silver which has the desired reflectivity. The inherent adhesion problem of silver was solved by utilizing an interface layer of Al_2O_3. Thicknesses, deposition rates, and substrate temperatures of both the silver and Al_2O_3 layers are all relatively critical factors affecting the mechanical stability and reflectivity and were, therefore, carefully controlled.
2. Silver reflectance may be "enhanced" in the near ultraviolet to give better solar reflectance. This enhancement was achieved in this work by the introduction of a near UV reflector stack consisting of alternate layers of TiO_2 and MgF_2. The

selection of these materials for the UV stack was based on several considerations. For example, due to the large differences in indices of refraction of the TiO_2 and MgF_2, the stack is broader than may otherwise be achieved with other materials (i.e., TiO_2 has perhaps the highest index of refraction for a near UV transparent material). Another advantage of this material combination is hardness of the materials, and excellent adhesion to the substrate.

The design of the stack was achieved with the aid of computer optimization to establish the minimum number of layers required to provide sufficient reflectivity. Fabrication simplicity was a major consideration.

3. A silver film is inherently soft and does not form a "protective oxide" on the surface exposed to the environment. In fact, Ag tarnishes thus being not only mechanically but also optically unstable without suitable protection. SiO was selected for the protective coating material and was found to satisfy the specifications. However, early in the program it was found that a layer of nichrome deposited prior to the SiO improved both the mechanical stability and overall performance of the mirror. As with most other materials, thickness and deposition rates required optimization to achieve sufficiently thick coatings without pinholes, crazing or blooming.

4. The deposition techniques utilized in all cases were vacuum evaporation or some variation of vacuum evaporation — using either boat or e-gun source heating as appropriate for the individual film materials and controlled partial pressures of O_2 to achieve desired stoichiometry. All three modes can be and were used simultaneously in our HV vacuum stations. These systems had, in all cases, deposition rate, thickness and substrate temperature controls. Thickness (and consequent rate) calibration was established via Talystep measurements of calibration samples deposited under each particular deposition condition of interest.

3.1 NON-ENHANCED REFLECTIVE COATING

It has been found that the required adhesion and reflectivity are satisfied by the following materials, procedures, and techniques:

1. Clean surface to be coated according to 3.1.1 below.
2. Deposit thin Al_2O_3 bonding layer to ~100 Å according to 3.1.2 below.
3. Deposit Ag reflective layer >1800 Å according to 3.1.3 below.
4. Deposit Nichrome (or Inconel) layer >450 Å according to 3.1.4 below.
5. Deposit SiO protective coating layer to ~4000 Å according to 3.1.5 below.

3.1.1 CLEANING PROCEDURE

Wash with 2 tbsp. alconox/gallon H_2O mixture for 5 minutes, ultrasonic.

Flush with deionized water.

Rinse with deionized water 2 minutes, ultrasonic.

Clean with 50 percent conc. HNO_3 solution — 2 minutes, ultrasonic.

Flush with deionized water.

Rinse with deionized water, 2 minutes, ultrasonic.

Wash with alconox mixture 15 minutes, ultrasonic.

Flush with deionized water.

Rinse with deionized water, 2 minutes, ultrasonic.

Rinse with reagent grade isopropanol, 2 minutes, ultrasonic.

With filtered air, dry to completion.

Mount on holder.

Use purified air for final dust removal prior to pumpdown.

3.1.2 DEPOSITION OF Al_2O_3 BONDING LAYER

Background pressure $< 2 \times 10^{-6}$ torr

Partial pressure of oxygen 5×10^{-5} torr

Deposition rate ~4 Å/sec

Thickness ~100 Å

Substrate temperature during deposition 300°C

Deposition material: single crystal optical quality boules of sapphire purchased from Union Carbide

E-gun deposition

3.1.3 DEPOSITION OF SILVER REFLECTIVE LAYER

Background pressure $< 2 \times 10^{-6}$ torr

Deposition rate ≥60 Å/sec

Thickness ≥1800 Å

Substrate temperature during deposition 80°C ±10°C

Deposition material: ROC/RIC*, Ag-01, powder 99.99% pure

R. D. Mathis S8A — 0.005 Ta boat or equivalent

3.1.4 DEPOSITION OF NICHROME LAYER

Background pressure $< 2 \times 10^{-6}$ torr

Deposition rate > 2.5 Å/sec

Thickness ≥ 450 Å

Deposition material: #26 nichrome wire purchased from Magnet Wire Supply Co., Chatsworth, California

R. D. Mathis S38 — 0.005 W boat or equivalent

3.1.5 PROTECTIVE COATING DEPOSITION — SiO

Background pressure $< 1 \times 10^{-6}$ torr

Deposition rate 10-14 Å/sec

Thickness ~4000 Å

Deposition material: Union Carbide, Select grade SiO, +10-3/8 in. mesh distributed by R. D. Mathis

R. D. Mathis SiO 0.10 W baffle box

Al_2O_3 and Ag are presently deposited in a single pumpdown utilizing an E-gun and one boat. The rates and thicknesses are controlled by a crystal monitor with an attached printout recorder. The source-to-substrate distance is presently 11 in. with a 15 in. source-to-crystal distance. The procedure is as follows: The substrates are chemically cleaned immediately before loading into the vacuum system. After loading, the system is pumped down to approximately 1×10^{-6} torr. Heating of substrates is then started. To thermally etch substrates, the temperature is taken up to approximately 400°C for 3/4 hour to 1 hour. The substrate temperature is then lowered to 300°C at which point the system is ready to start the deposition. A partial oxygen pressure of 5×10^{-5} torr is introduced into the chamber. The deposition rate of the Al_2O_3 is brought to the specified value before the shutter is opened. After the rate stabilizes, the shutter is opened for a controlled period of time (e.g., 25 seconds for a rate of 4 Å/sec). After the Al_2O_3 deposition, the O_2 supply is shut off and the chamber pressure returns to 2×10^{-6} torr. The substrate heaters are then turned down to a stabilized substrate temperature of 80°C (requiring a wait of approximately 60 minutes) for the silver deposition. The silver source is then heated to produce a rate of >60 Å/sec, and the shutter opened for approximately 30 seconds.

* Research Organic/Inorganic Chemical Corp., Sun Valley, California

The substrates are then allowed to cool to <40°C, requiring about 40-50 minutes and immediately thereafter transferred to a second system for the nichrome and SiO protective coating depositions. SiO was selected for the protective coating material, and, based on tests performed to date, appears to satisfy the specifications.

The deposition procedure involves pumpdown to 1×10^{-6}, at which time the nichrome source is brought to the desired rate and deposited for the time required to give the desired thickness. This is followed by the heating of the SiO source (no partial O_2 pressure added) in a SiO 0.10 W (tungsten) baffle box to the desired rate (10-14 Å/sec), opening the shutter for time interval so as to deposit ~4000 Å of the SiO, closing the shutter and allowing the system to cool. No substrate heating is utilized for these depositions.

The depositions were performed, utilizing appropriate substrate holders, such that the substrate was covered completely over the top surface and edges and around the circumference.

3.2 ENHANCED REFLECTIVE COATING

The enhanced second surface reflectivity mirror consists of the following coatings and order of deposition:

1. Reflectance stack for near UV enhancement (TiO_2 + MgF_2 stack).
2. Adhesion and reflectivity coating for λ from ~3500 Å into the IR (Al_2O_3 + Ag).
3. Protective coating (Nichrome + SiO).

Reflectivity Stack for UV Enhancement

The near ultra violet reflectivity enhancement has been implemented by a computer optimized TiO_2/MgF_2 reflectivity stack of the following design:

Quartz Substrate	Layer #	Thickness (Å)	Rate
TiO_2	1	152	8 Å/sec
MgF_2	2	544	30 Å/sec
TiO_2	3	288	
MgF_2	4	599	
TiO_2	5	259	
MgF_2	6	585	

Quartz Substrate	Layer #	Thickness (Å)	Rate
TiO_2	7	307	
MgF_2	8	576	
TiO_2	9	342	
MgF_2	10	457	

The TiO_2 source material was purchased with 99.99% purity and the MgF_2 was purchased with 99.999% purity.

The deposition of TiO_2 utilized an E-gun while the MgF_2 is deposited with an 0.005 molybdenum boat. The rate and thickness are controlled by a crystal monitor again with an attached printout recorder. The procedure is as follows: the substrates are chemically cleaned utilizing the same procedure adopted for the unenhanced substrates (i.e., described in Section 3.1.1). Immediately after cleaning, they are loaded into the vacuum system. After loading, the system is pumped down to less than 1×10^{-6} torr. The substrates are then heated, first to approximately 400°C for thermal etching, after which the temperature is lowered to 250°C for the deposition of the stack. The first layer is TiO_2 in which case the deposition rate of the TiO_2 is brought to the specified value before the shutter is opened.

The deposition of the TiO_2 takes place in a partial pressure of 3×10^{-5} torr of oxygen. The shutter is opened for the length of time required to deposit the specified thickness of TiO_2 at the specified rate. At the completion of the TiO_2 deposition, the O_2 is pumped out until the chamber pressure is $<1 \times 10^{-6}$ torr. The boat containing the MgF_2 is then heated and the deposition made in the manner just described. The procedure is the same for all TiO_2 and MgF_2 layers in the stack. Since this stack is designed with modified thicknesses throughout (modified quarter-wave), it is essential to accurately control these thicknesses. At the completion of the stack, the system is cooled down to <50°C (in approximately 100 min), and then opened to add the Al_2O_3 and Ag (without disturbing substrates) for the adhesion and reflectance coatings. Although, for the depositions performed to date, it was necessary to open the system in between the deposition of the stack and the Ag reflectance coating (since a single source E-gun was used), it is recommended that these depositions be performed, in the future, in a single pumpdown by using a multiple source E-gun. After loading the system with the Al_2O_3 and Ag, the system is again pumped to less than 1×10^{-6} torr for the deposition of the adhesion and reflectance coating.

Adhesion and Reflectance Coating

These depositions are identical to the corresponding depositions for the unenhanced mirror (see Sections 3.1.2 and 3.1.3).

Protective Coating

These depositions are identical to the corresponding depositions for the unenhanced mirror (see Sections 3.1.4 and 3.1.5).

SECTION 4

FEP TEFLON EXPERIMENTS

A flexible second surface mirror, FEP Teflon, coated on the back surface with silver (generally referred to as silver Teflon) has found considerable use on satellites. It may be applied to curved surfaces, is reasonable in cost, and has performance characteristics only slightly degraded from the fused silica second surface mirrors.

Although FEP Teflon degrades under space environmental conditions, the application is expected to continue for some time. Silver Teflon on a flat plate is essentially a specular reflector; making it a diffuse reflector would allow its use on certain advanced satellites. A silver Teflon modified to provide diffuse reflectance was selected as one of the four program candidate surfaces for development and testing.

Various methods of surface contouring were tried, including spraying an FEP Teflon liquid onto a contoured surface, powder fusion, abrasion of Teflon film, and replication between two contoured plates.

4.1 SPRAY FORMING

It was found that a Teflon film of good appearance could be formed by spraying an FEP Teflon spray onto a roughened surface and thermal curing the film. The resulting film material was clear and apparently gave good diffusion and transmission characteristics, however, after extensive discussion with DuPont representatives, it was determined that there are, in addition to the FEP Teflon, certain solvents and solubilizing agents in the spray. Since there was no reliable way to predict space stability of these diluents without extensive space testing and since the amount of effort that could be allotted to this portion of the program was limited, it was elected to pursue a different approach.

4.2 POWDER FUSION

Some work was done with powder fusion. There were problems in outgassing of the resultant films and methods of forming a uniform film and controlling its thickness were not readily available. "Spooning out" the material and compressing between two plates was not satisfactory. A feasible process for producing a contoured Teflon film may exist, but the replication method described below accomplished the same result with less effort and the powder fusion method was not extensively pursued.

4.3 ABRASION

Abrasion: grinding, honing, sanding, sand blasting, etc., when applied to FEP Teflon at room temperature resulted in embedded grit and lose debris covering the surface Cleaning the surface was difficult.

Optical performance of the Teflon after abrasion was very poor. At cryogenic temperatures, the Teflon does become hard and possibly some abrasive techniques might work if the material were cooled, but it was not apparent how a microscopically smooth surface could be obtained from a surface abraded at cryogenic temperatures.

4.4 REPLICATION

It was found that good contoured FEP Teflon films could be made by pressing and heating a thin sheet (0.010-in. material was used) of commercially available FEP Teflon between two contoured glass or quartz molds. Molds were made of 1/4"-thick glass and quartz just as the fused silica second surface mirror substrates were made, i.e., by grinding and etching. A sandwich was made, with the Teflon sheet between the contoured surfaces of the two molds. Upon application of vacuum, heat, and pressure, the Teflon very closely replicated the contoured molds.

4.4.1 CONTOURED MOLDS. Attempts were made to make one Pyrex* mold with a 3 μm ground and HF etched surface and one mold with a 9 μm ground and etched surface.

The procedures used did not produce a uniformly contoured surface, rather there were miscellaneous large kidney shaped gouged impressions in the relatively uniform contoured surface. This problem was at first attributed to the use of Pyrex that had been used in making the earlier FEP Teflon diffuse sheets. The heating of the Pyrex was thought to have caused some crystallization which led to non-uniform etching. New, virgin Pyrex was procured and grinding and etching performed. The same kidney-shaped, gouged impressions appeared.

Two quartz blanks were obtained and ground and etched. One of these blanks was ground out with 30 μm grit on one side and 3 μm on the other. The other blank was ground out with 30 μm on one side and 9 μm on the other. The blanks were ground by two different people (optics technician and optical designer). Interestingly, the 3 μm side of the 30 μm-3 μm blank was perfect, the 30 μm side had only a very few kidney-shaped impressions. The 9 μm-30 μm blank had a large number of kidneys on both sides.

After some consideration, the conclusion was reached that the kidneys were due to deep scratches or cracks in the blanks; the HF apparently, rather rapidly "cleans" out all scratches or cracks. If the scratch goes deep, a deep and broad kidney results.

*Trademark, Corning Glass Co.

If the scratches and cracks are shallow and relatively uniform, a uniformly contoured surface results. If this was the explanation of difficulties, the solution was obvious. Thoroughly "grind out" the surface to be etched with the final grit. Apparently in grinding the surfaces, one man had been more thorough than the other.

In making mirrors, windows, and lenses, it has always been the practice to start grinding with a coarse grit and to use successively finer grits and finally a very fine grit as a polish. If one changes grits too soon, the final polishing will reveal one's error (haze and scratches that will not polish out) and regrinding is indicated.

In the case at hand, a thorough grinding at the final grit was indicated; when this was done, the HF etch produced a relatively uniform contoured surface.

4.4.2 RELEASE AGENT. Teflon, heated to 550°F or so, makes a glue of almost unparalleled excellence. The Pyrex and quartz molds with Teflon in between after heating could not be separated. A release agent was used which, when applied to the molds, served to prevent the Teflon from sticking. The treated molds could be reused many times without recoating. In fact, it was required to use them several times to remove any excess release agent. The agent was a commercial product called Frekote 33.[2] The label on the can gave no hint of the nature of the material.

4.4.3 REPLICATION PROCESS. It was found that when a 4-in. × 4-in. Teflon sheet was placed between Pyrex molds in an ordinary oven at about 500 to 600°F, a replication of the surface of part of the Pyrex plates onto the Teflon occurred. Gas bubbles occur to some extent between the plates on the Teflon to prevent replication over the entire surface. This problem was overcome by use of a vacuum bagging technique which is in common usage in certain types of graphite/epoxy composite material manufacture (see Figure 4-1).

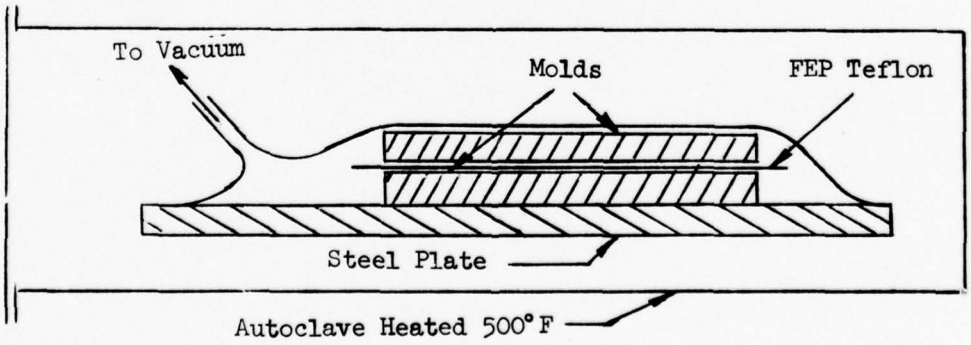


Figure 4-1. Vacuum Bagging Arrangement

The steps in this bagging technique are as follows:

1. The film and two molds are assembled.
2. The vacuum bag is prepared by:

a. Locating the mold-Teflon assembly on the steel base plate, as shown in Figure 5-1.

b. Placing silicone adhesive on periphery of assembly.

c. Attaching vacuum line.

d. Packing corners with porous glass cloth to smooth the package.

e. Applying a silicone blanket separated by H-film (Kapton) from the quartz molds.

f. Firmly pressing the silicon seal at the periphery to achieve vacuum tightness.

g. Inserting in the assembly in an oven.

3. The assembly is soaked in the oven with a vacuum in the bag under the following conditions:

   a. Temperature — 550°F (288°C)

   b. Time at temperature — 45 minutes

   The temperature schedule may be manual or programmed.

4. The quartz plates are cooled to room temperature before the Teflon film is removed to keep the Teflon sheet from crinkling.

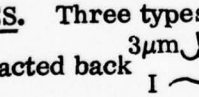
SECTION 5

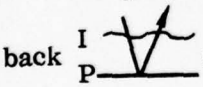
SELECTION OF CANDIDATES

Four candidates were to be selected for complete evaluation. To arrive at the designs from which these candidates would be selected, theoretical and empirical ideas were tried. Simply put, this involved "make it and test it." Testing involved the quick look optical tests described in Section 2.2. Pursuing this approach, it developed that the grinding and etching method was to be preferred for forming fused silica blanks and that the replications of commercial FEP Teflon sheet was preferred for Teflon contouring. Having established that these methods would be used, the varieties of grit size, times of etch, and for Teflon, time and temperature in the autoclave were varied. Four general categories of samples selected and multiple substrates for designs within categories were made.

5.1 CATEGORIES

5.1.1 GROUND AND ETCHED 3 μm FRONT-9 μm BACK. In this category, the time of etching was varied. Six or more samples were etched for the following times: 1, 1-1/2, 2, 2-1/2 hours.

5.1.2 ULTRASONIC IMPACTED SAMPLES. Three types of ultrasonic impacted samples were prepared: 3 μm front, impacted back 

; impacted front, polished back 

; and impacted front, 3 μm back 

. The impact tool face was a copper disc, silver soldered to the impact head. A spiral pattern was cut in the copper with a rounded tool. A cross-section of the spiral is given in Section 2.8.

It was noted in viewing impacted samples under a microscope that the grooves tended to become perceptibly shallower after about six or seven samples were made with a new tool. As an operating procedure the first six samples made with a new tool were categorized "deep," the second six shallow. The tool was refaced after manufacture of 12 samples.

5.1.3 GROUND AND ETCHED FRONT SURFACE, POLISHED BACK SURFACE. In this category etch time and grit size were treated as variables:

Grits: 3 μm, 9 μm, 25 μm, and 30 μm, and
Times: 1/4, 1/2, 3/4, 1, and 2 hours

5.1.4 FEP TEFLON. Both Pyrex and quartz molds were used to replicate the etched pattern onto Teflon sheet. Three Teflon categories were tested. The coded sample designations and explanations of the codes are as follows:

FEP/3-9/P/.25/GTS

Material: 0.010-in. thick FEP Teflon

Top mold contour: 3 μm grind, HF etch

Contour bottom (silvered side) mold: 9 μm grind, HF etch

Mold material: Pyrex

Mold etch time: 0.25 hr

Silvered: by Sheldahl

FEP/3-9/Q/.83/GTS

As above except:

Mold material: quartz

Mold etch time: 0.83 hr

FEP/30-p/P/.5/GTS

As in first sample above except:

Top contour mold: 30 μm grind, HF etch

Contour bottom mold: Polished surface

Mold etch time: 0.5 hr

In these three categories, "test silver"* was applied to the substrates to allow determination of "quick look" data. Sheldahl, under subcontract, applied the silver to samples for complete evaluation.

5.2 QUICK LOOK RESULTS

Table 6-1 indexes the quick look data on the above described four categories. These data were used to select promising candidates for complete evaluation. The principal criteria considered were low bidirectional reflectance and low solar absorptance. Secondary considerations were cost and producibility.

*"Test silver" coatings made by evaporative application of silver without a preliminary layer to enhance adhesion and no overcoating for mechanical and environmental protection.

TABLE 5-1
INDEX TO QUICK LOOK TABLES

Category	Type of Surface	Hours Etch	Bidirectional Reflectance $\theta = \theta' = 45°$	Bidirectional Reflectance $\theta = \theta' = 20°, 30°, 40°, 50°, 60°, 70°, 80°$	Directional-Hemispherical Reflectance λ - 0.4, 0.45, 0.5, 0.55 μm	Table Number
1. Ground Silica	3μm- 9μm	1.0	X		X	5 - 2
	3μm- 9μm	1.5	X		X	5 - 3
	3μm- 9μm	2.0	X		X	5 - 4
	3μm- 9μm	2.5	X		X	5 - 5
	3μm- 9μm	1.0, 1.5, 2.0, 2.5		X		5 - 6
	3μm- 9μm	1.0, 1.5, 2.0, 2.5	X			5 - 1 Graph
2. Impacted Silica	3μm- Impact Ground	1.5, 2.0, 2.5	X		X	5 - 7
	3μm- Impact Ground	2.0		X		5 - 8
	Impact Ground- Polished	1.0, 1.5 2.0, 2.5	X		X	5 - 9
	Impact Ground- 3μm	2.0, 2.5	X		X	5 - 10
	Impact Ground- 3μm	2.0, 2.5		X		5 - 11
3. Ground-Polished Silica	9μm- Polished	0.25, 0.5, 0.75, 1.0, 2.0	X		X	5 - 12
	25μm- Polished	0.25, 0.5, 0.75, 1.0, 2.0	X		X	5 - 13
	30μm-Polished	0.5, 1.0, 1.5, 2.0, 2.5	X		X	5 - 14
	9μm, 25μm, 30μm, Polished	0.25, 0.5 1.0		X		5 - 15

Table 5-1 (Continued)

Category	Type	Hours Etch	Bidirectional Reflectance $\theta = \theta' = 45^\circ$	Bidirectional Reflectance $\theta = \theta' = 20^\circ, 30^\circ, 40^\circ, 50^\circ, 60^\circ, 70^\circ, 80^\circ$	Directional-Hemispherical Reflectance λ - 0.4, 0.45, 0.5, 0.55 μm	Table Number
4. FEP Teflon	3μm& 9 μm Pyrex Plates	0.25	X	X	X	5 - 16
	3μm& 9 μm Quartz Plates	0.83	X	X	X	5 - 16
	30 μm& Polished Pyrex Plates	0.5	X	X	X	5 - 16
	FEP Plain Sheet	0	X	X	X	5 - 16

TABLE 5-2
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 314 Fused Silica	1.0	3 μm 9 μm	1.5	.943	.953	.953	.954
SS 315 Fused Silica	"	"	1.4	.943	.953	.958	.954
SS 316 Fused Silica	"	"	1.5	.943	.958	.958	.959
SS 317 Fused Silica	"	"	1.4	.943	.953	.953	.959
SS 319 Fused Silica	"	"	1.4	.943	.953	.958	.959
SS 320 Fused Silica	"	"	1.4	.938	.948	.953	.954
Average			1.43	.942	.953	.955	.956
Average of Averages				.952			

TABLE 5-3
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 288 Fused Silica	1.5	3 μm 9 μm	2.0	.949	.964	.963	.965
SS 289 Fused Silica	"	"	2.1	.949	.959	.963	.965
SS 290 Fused Silica	"	"	1.7	.943	.959	.958	.960
SS 291 Fused Silica	"	"	1.7	.943	.953	.953	.955
SS 292 Fused Silica	"	"	1.7	.943	.959	.963	.965
SS 293 Fused Silica	"	"	1.8	.949	.964	.963	.960
SS 294 Fused Silica	"	"	1.8	.943	.959	.958	.960
SS 295 Fused Silica	"	"	1.8	.949	.964	.963	.965
Average			1.82	.946	96	960	961
Average of Averages				.956			

*ρ′ = Bidirectional Reflectance θ = θ′ = 45° (in plane), Units: Steradians^{-1}

TABLE 5-4
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 297	2.0	3μm 9 μm	2.4	.948	.964	.969	.965
SS 298	"	"	2.2	.948	.964	.969	.965
SS 299	"	"	2.2	.948	.959	.963	.965
SS 300	"	"	2.0	.948	.959	.963	.960
SS 301	"	"	2.2	.938	.948	.953	.965
SS 302	"	"	2.2	.943	.959	.963	.960
SS 303	"	"	2.2	.948	.969	.969	.970
SS 304	"	"	1.9	.948	.959	.963	.965
Average			2.16	.946	.960	.964	.964
Average of Averages				958			

TABLE 5-5
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 306	2.5	3 μm 9 μm	2.7	.949	.959	.963	.970
SS 307	"		2.4	.953	.969	.969	.970
SS 308	"		2.2	.938	.959	.963	.965
SS 309	"		2.4	.949	.969	.969	.970
SS 310	"		2.3	.953	.964	.969	.965
SS 311	"		2.3	.959	.969	.969	.970
SS 312	"		2.7	.959	.969	.969	.970
Average			2.43	.951	.965	.967	.969
Average of Averages				.963			

*ρ′ = Bidirectional Reflectance θ = θ′ = 45° (in plane), Units: Steradians^{-1}

TABLE 5-6
QUICK LOOK DATA
FUSED SILICA

Sample	Hrs. HF Etch	Configuration	In Plane Bidirectional Reflectance at Specular Angle ($\theta = \theta'$), λ = .5 μm							
			20°	30°	40°	45°	50°	60°	70°	80°
SS 317	1	3 μm / 9 μm	.96	1.09	1.15		1.6	2.4	5.4	6.1
SS 295	1.5	"	1.3	1.4	1.6		1.9	3.3	6.9	8.6
SS 298	2	"	1.6	1.7	2.1		2.4	4.0	7.7	8.8
SS 307	2.5	"	1.8	2.1	2.3		2.8	4.3	9.2	7.7

TABLE 5-7
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ =			
				.4 μm	.45 μm	.5 μm	.55 μm
SS 347 deep	1.5	3 μm / I	1.0	.830	.938	.942	.944
SS 352 shallow	1.5	"	.76	.911	.938	.938	.939
SS 348 deep	2	"	1.1	.932	.943	.953	.955
SS 353 shallow	2	"	1.1	.943	.953	.953	.960
SS 349 deep	2.5	"	1.0	.917	.932	.938	.933
SS 354 shallow	2.5	"	1.6	.943	.953	.958	.960

TABLE 5-8
QUICK LOOK DATA
FUSED SILICA

Sample	Hrs. HF Etch	Configuration	In Plane Bidirectional Reflectance at Specular Angle ($\theta = \theta'$), λ = .5 μm							
			20°	30°	40°	45°	50°	60°	70°	80°
SS 348 deep	2	3 μm / I	.76	.84	1.08	1.54	1.54	2.68	6.5	7.1
SS 353 shallow	2	"	.91	1.1	1.38	1.67	1.9	3.18	7.1	7.6

*ρ′ = Bidirectional Reflectance $\theta = \theta' = 45°$ (in plane), Units: Steradians^{-1}

TABLE 5-9
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 337	1		3.5	.954	.969	.968	.970
SS 338	1	"	4.8	.948	.953	.963	.970
SS 331	1.5	"	5.0	.948	.964	.968	.970
SS 332	1.5	"	6.5	.959	.970	.974	.975
SS 333	2	"	6.1	.953	.963	.969	.965
SS 334	2	"	7.4	.964	.974	.974	.980
SS 335	2.5	"	6.4	.948	.963	.969	.970
SS 336	2.5	"	7.4	.964	.974	.974	.975

TABLE 5-10
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 350	2		2.2	.953	.970	.970	.970
SS 351	2.5	"	2.5	.953	.970	.970	.970

TABLE 5-11
QUICK LOOK DATA
FUSED SILICA

Sample	Hrs. HF Etch	Configuration	In Plane Bidirectional Reflectance at Specular Angle (θ = θ′) λ = .5 μm 20°	30°	40°	45°	50°	60°	70°	80°
SS 350	2		1.7	1.7	2.0	2.2	2.2	2.5	2.9	1.9
SS 351	2.5		2.1	2.2	2.3	2.5	2.7	2.6	3.3	1.7

*ρ′ = Bidirectional Reflectance θ = θ′ = 45° (in plane), Units: Steradians^{-1}

TABLE 5-12
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 268	.25	9μm P	3.2	.948	.958	.958	.959
SS 269	.25	"	3.6	.943	.958	.958	.959
SS 270	.5	"	4.6	.954	.969	.969	.970
SS 271	.5	"	5.1	.959	.969	.969	.970
SS 272	.75	"	6.5	.959	.969	.974	.975
SS 273	.75	"	6.7	.964	.974	.974	.975
SS 274	1.0	"	6.1	.959	.974	.974	.975
SS 275	1.0	"	6.7	.964	.974	.979	.98
SS 279	2.0	"	6.7	.970	.979	.979	.98

TABLE 5-13
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 246	.25	25μm P	1.9	.932	.948	.953	.960
SS 247	.25	"	1.9	.922	.943	.948	.950
SS 248	.50	"	2.6	.938	.953	.958	.965
SS 249	.50	"	2.8	.943	.959	.958	.960
SS 250	.75	"	3.8	.949	.959	.963	.965
SS 251	.75	"	3.4	.943	.959	.963	.960
SS 253	1.0	"	4.0	.949	.964	.963	.965

*ρ′ = Bidirectional Reflectance $\theta = \theta' = 45^{o}$ (in plane), Units: Steradians^{-1}

TABLE 5-14
QUICK LOOK DATA
FUSED SILICA

Sample No. and Description	Hrs. HF Etch	Configuration	*ρ′	Dir.-Hem. Reflectance at λ = .4 μm	.45 μm	.5 μm	.55 μm
SS 258	.5	30 μm P	1.8	.932	.948	.953	.955
SS 259	.5	"	1.7	.932	.948	.953	.955
SS 261	1.0	"	2.4	.938	.953	.958	.960
SS 262	1.5	"	2.8	.943	.958	.958	.960
SS 264	2.0	"	3.6	.943	.958	.963	.970
SS 265	2.0	"	3.9	.953	.969	.969	.970
SS 266	2.5	"	3.2	.943	.958	.963	.965

TABLE 5-15
QUICK LOOK DATA
FUSED SILICA

Sample	Hrs. HF Etch	Configuration	In Plane Bidirectional Reflectance at Specular Angle (θ = θ′) λ = .5 μm 20°	30°	40°	45°	50°	60°	70°	80°
SS 268	.25	9 μm P	3.6	3.4	3.4	3.3	3.2	3.0	2.9	5.2
SS 247	.25	25 μm P	2.0	2.0	2.0	2.0	1.8	1.9	2.6	12.0
SS 249	.50	"	3.3	3.1	2.9	2.9	2.8	2.6	2.6	18
SS 258	.50	30 μm P	1.8	1.9	1.9	1.9	1.9	1.8	1.6	11
SS 261	1.0	"	2.5	2.4	2.3	2.4	2.3	2.3	2.1	12

*ρ′ = Bidirectional Reflectance θ = θ′ = 45° (in plane), Units: Steradians^{-1}

TABLE 5-16
QUICK LOOK DATA
FEP TEFLON

Sample*	Directional Reflectance at λ =				In Plane Bidirectional Reflectance* at Specular Angle (θ = θ) λ = .5 μm							
	.4 μm	.45 μm	.5 μm	.55 μm	20°	30°	40°	45°	50°	60°	70°	80°
FEP, 3-9, .25 Pyx, GTS, #1	.722	.813	.855	.882	2.5	2.9	2.8	2.9	3.6	3.7	6.4	15
FEP, 3-9, .25 Pyx, GTS, .25	.757	.823	.860	.882	2.7	2.9	2.8	2.7	3.0	3.6	7.4	14.4
FEP, 3-9, .83 Qtz, GTS, #1	.775	.860	.891	.908	2.1	2.3	2.1	2.6	2.5	3.8	9.8	11.3
FEP, 3-9, .83 Qtz, GTS, #2	.759	.844	.881	.898	2.0	2.3	2.5	2.3	2.7	4.1	9.9	13.3
FEP, 30-P, .5 Pyx, GTS, #1	.827	.891	.912	9.29	18	18	17	11	13	10	9	8
FEP, 30-P, .5 Pyx, GTS, #2	.812	.875	.907	.924	15	15	14	10	11	8	9	9
FEP plain sheet	.896	.922	.948	.955								

FEP = FEP Teflon

3 - 9 = 3 μm mold used on one side 9 μm mold used on other side. 9μm side silvered.

Pyx = Pyrex glass mold

.25 = 0.25 hr etch of mold

GTS = Silver applied by Sheldahl

#1,etc= Sample number of particular type

.83 = 0.83 hr etch of mold

Qtz = Quartz molds

30 - P = Front side contoured with 30 μm mold

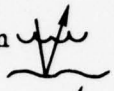
.5 = 0.5 hr etch of mold

* Units: Steradians^{-1}

5.3 DISCUSSION OF RESULTS

5.3.1 3 μm FRONT-9 μm BACK. This design provides high reflectance and is a very diffuse reflection from small to moderately large incidence angles. For this design a tradeoff between low bidirectional reflectance and low solar absorptance was possible. This may be understood by reference to Figure 5-1 where an "average of the average" reflectance at 0.4, 0.45, 0.5, and 0.55 μm is plotted against the bidirectional reflectance. From this 3 μm-9 μm category two designs were selected for complete evaluation: mirrors which have been etched 1.5 and 2.5 hr.

5.3.2 IMPACTED DESIGNS. Two of the designs in this category showed very good performance:

- 3 μm front; impacted back 

- impacted front; 3 μm back 

The second design (impacted front) had a particularly important property; a nearly constant (and moderate) bidirectional reflectance as a function of incidence angle was obtained up to very large incidence angles. This property could prove useful in the design of a cover slip for a diffusely reflecting solar cell. It should provide highly diffuse reflectance irrespective of the angle of incidence of sunlight and might well increase the performance of the cell at off-normal light incidence angles.

5.3.3 GROUND AND ETCHED FRONT SURFACE–POLISHED BACK SURFACE. This design provided good diffuse reflectance performance as a function of angle of light incidence and one type in this category was selected for complete evaluation (0.5-hr etch).

Increasing etch time increases reflectance, however, this also increases the bidirectional reflectance. This relationship allows a mirror to be tailored to a particular need. By selection of etch time, the optimum performance for a particular application may be selected.

5.3.4 FEP TEFLON. Using quartz molds, contoured FEP Teflon with satisfactory diffuse reflectance performance was obtained (mold etch time 0.83 hr) but the reflectance was unsatisfactory. It is highly likely that with more development effort the reflectance performance of this material can be improved to essentially equal the performance of the plain silvered FEP sheet. In this program the fused silica substrates were given priority.

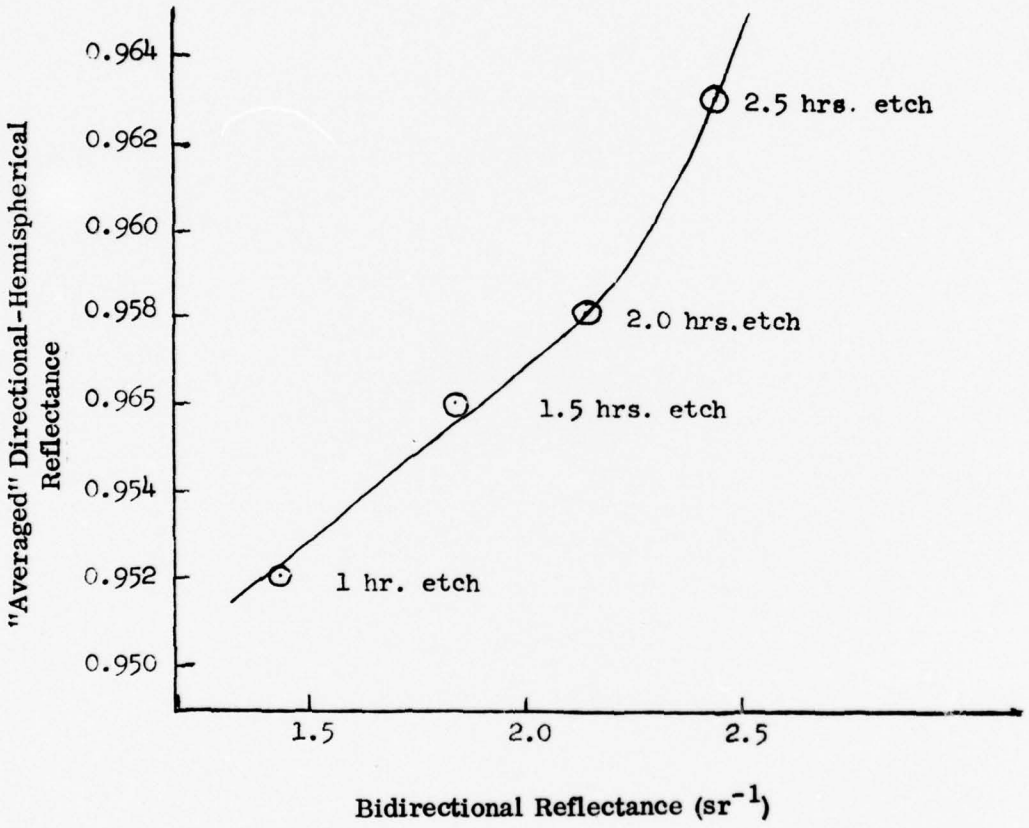


Figure 5-1. Directional-Hemispherical Reflectance of 3 μm-9 μm Samples Plotted Against Bidirectional Reflectance (at Specular Angle for Incidence Angle of 45°) for Various Etch Times

SECTION 6

FABRICATION RECIPES

The methods of substrate manufacture are described in general terms in Sections 2.0 and 4.0. In this section explicit manufacturing methods are provided for each of the four types of second surface mirrors developed.

6.1 FUSED SILICA GROUND AND ETCHED BOTH SIDES

Corning 7940 fused silica blanks, $1 \times 1 \times 0.040$-in. thick, unpolished are available and are a convenient starting wafer. Mark Optics (Santa Ana, CA) and probably other optical supply houses can supply these in one to two weeks time. Ten of these squares are pitched down to an aluminum tool about 5 in. in diameter and flat to within 3 or 4 fringes. The samples are generated to make their surface parallel and attain the proper starting thickness. The surfaces are ground and etched according to sequences shown in Table 6-1. The substrate grinding and polishing operations are routine for a good optics technician and the terms "generate," "pitch down," "grind out," and "felt polish" are commonly used by these technicians. The thicknesses in the schedule are not sacred; other grinding schedules may arrive at the same end wafer. The schedule given provides one way to reliably obtain the desired substrates. The need for a large number of wafers might well justify some attention to simplified production techniques. The diffused second surface mirror should be competitive in cost with the current commercial specularly reflecting items.

Table 6-1. Grinding-Etching Schedule for 3μ-9μ Diffuse Mirrors

Operation on Wafer	Resultant Thickness	
	1.5 hr HF Etch	2.5 hr HF Etch
Pitch to tool	0.040	0.040
Generate to make parallel	0.028	0.030
Grind with 25 μm grit	0.024	0.026
Grind with 9 μm grit	0.022	0.024
Remove from tool and repitch fine ground (9μ) against tool		
Grind with 25 μm grit	0.018	0.020
Grind with 14 μm grit	0.016	0.018
Grind with 3 μm grit	0.014	0.016
Etch with 2:1 HF Solution for time shown in column heading	0.010	0.010

Difficulties encountered in the process of developing the substrate:

1. Uneven etch of the surface to give visible blotches. This problem is more cosmetic than real. Samples with blotches performed as well as the pretty samples. Insufficient cleaning before etching may have caused this blotched appearance, although internal strains in the fused silica can cause variations in the etching rate and thus the appearance. In any event, improper cleaning before etching (leaving pitch residue) can cause uneven etching. Cleaning with solvents and then hot cleaning solution (until solution wets entire wafer) completely removed contaminants which could influence etching rates.

2. Inadequate "grind out" with grinding compounds becomes painfully evident after etching. Generation marks and scratches show up clearly. (Some optics houses use a very mild etch to make evident inadequate grind out.) Throw away samples which reveal generator marks (they will be too thin to rework) and start over with a resolve to thoroughly grind out the surface with each grit size.

3. The grinding compound tends to break down and become a fine grit if it is not constantly renewed. The "bowl feel" technique for low scatter mirror is based on this phenomenon. In this work, the grind compound should be constantly renewed.

4. Without some sort of code, the question "which side is up" on the 3 μm-9 μm samples will occur during manufacture. In this work one corner was ground in a non-symmetrical fashion to distinguish the 3 μm and 9 μm sides.

6.2 FUSED SILICA, ETCHED ONE SIDE, POLISHED ONE SIDE

Table 6-2 provides necessary information for manufacture of the 30 μmP samples. Most comments under 6- 1 above also apply here.

6.3 RELATIVE COST

In production quantities, it is likely that the cost of these diffuse mirrors would be no more than the cost of the specular commercial mirrors. The grinding and etching process should be accomplishable at a cost no greater than the polishing cost for the commercial mirrors. Actually, the cost of fabricating the diffuse substrate might be less than the cost for the specular substrate.

6.4 FEP TEFLON

The forming of contoured FEP Teflon is discussed in some detail in Section 4. Only a tentative recipe for fabrication will be given here because, although a promising approach was developed, further work is needed to obtain higher performance material.

Molds of about 4-in. square and 1/2-in. thick should be generated smooth, ground, and polished on the sides to be used for replication. One polished plate should then be

lapped with 3 μm grit, and the other with 9 μm grit. The object of polishing and then grinding is to assure that thoroughly "ground out" 3 μm and 9 μm surfaces are obtained. Etch the plates for 50 minutes in 2:1 HF solution. Cover the 3 μm and 9 μm surfaces of the plates with Frekote S3 release agent. Wipe off as much of this agent as possible with a soft cloth. Use the molds through six cycles of forming FEP Teflon without the bagging procedure, i.e., place FEP Teflon sheet between plates, place the plates in a

Table 6-2. Grinding Schedule for 30 μm Polished Diffuse Mirrors

Operation on Wafer	Resultant Thickness (in.)
Pitch to tool	0.040
Generate to make parallel	0.026
Grind with 30 μm grit	0.020
Remove from tool, clean thoroughly, etch for 0.5 hr in 2:1 HF solution (both sides)	
Pitch 30 μm and etched side against tool	
Grind with 25 μm grit	0.014
Grind with 14 μm grit	0.012
Grind with 3 μm grit	0.010+
Felt polish	0.010

550°F oven for 45 minutes, remove from oven and after cooling, separate plates, discard the Teflon sheet, and repeat the process six times.* Next, prepare a production run using bagging. The bagging procedure is described in Section 4.

*Difficulties with the samples of 3 μm-9 μm FEP made under this program are attributed to residual release agent on the molds. The agent presented problems in cleaning prior to silvering; in applying several cleaning methods, the quality of the soft replicated surface was degraded. The use of the molds through several cycles does eliminate the problem of residual release agent; in earlier experiments in which molds were used many times, contoured Teflon that was bright and clear was obtained. These early molds produced Teflon in which the contouring was too coarse for good optical performance; the five contoured quartz molds apparently were used too few times to remove the excess release agent. This is one area where further work is recommended.

SECTION 7

CHARACTERIZATION OF SURFACES

Ideally, to characterize a surface is to give a mathematical expression of its shape. In this case, however, the complexity of the surfaces resulting after grinding and etching, as evidenced by the photographs shown in Figure 7-1, does not readily permit the derivation of an exact expression for the surface. It was possible, however, to experimentally determine the general characteristics of these surfaces; top view and profile cross-section pictures were obtained.

Top View

Scanning electron microscope photographs of representative ground and ground and etched fused silica wafers were obtained. These are shown in Figure 7-1. The photographs are shown in order of increasing grit size from left to right (3, 9, 22, 177 μm) and increasing time of etch from top to bottom. The top picture in each column represents an unetched sample followed in order by etch times of 0.5, 1, 2 and 2.5 hours. All photographs were taken at the same magnification, 300X. Thus, a segment of 15 mm on the photograph is equivalent to actual spacing on the wafer of 50 μm.

Profiles of the Surface

A variety of methods were tried in obtaining the surface profile. These included scans with the Schmaltz Profile Microscope, the Nomarski attachment to a microscope and a mechanical profilometer. None of these was satisfactory.

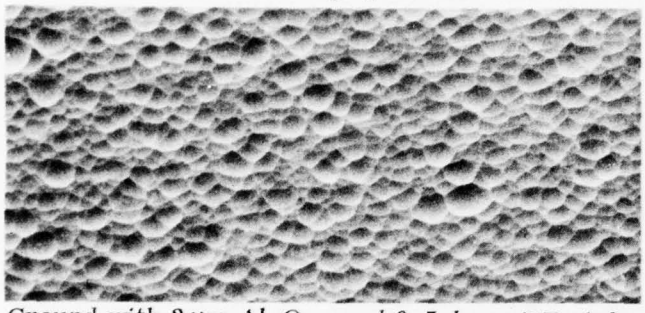
The Nomarski attachment, being a double beam shearing interferometer, could only give the size of the cells relative to the neighboring ones and not in absolute units. This would have been acceptable if a neighboring area were flat to serve as a standard, which was, of course, not the case. The attachment did help to establish that the depressions produced after the etching are quite shallow and rather flat (crater-like), in the greater majority of cases. This was determined by counting the number of fringes produced for each region, and comparing them with those of their neighbors.

The profilometer seemed to be the ideal equipment for this job. It consists of a conically shaped needle which rides on the surface of the material in a given direction; the resulting vibrations of the needle are then analyzed and plotted by electronic equipment. After several trials and some arithmetic, it was clear that the variations of the surface are well beyond the mechanical capabilities of the machine. The needle used originally had a radius at the tip of 0.0005 in., which in the scale of the photographs of Figure 7-1 is represented by a circle of diameter of 0.3 in. (7.6 mm) (see Figure 7-3).

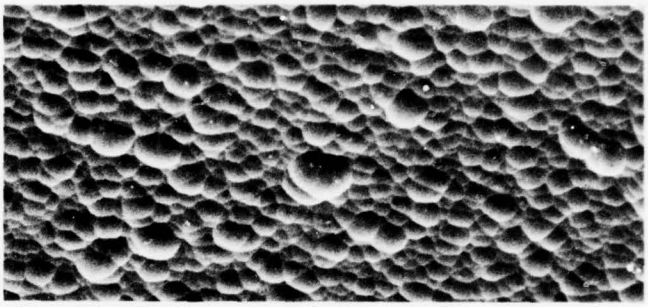
Ground with 3 μm Al_2O_3, no etching

Ground with 9 μm Al_2O_3, no etching

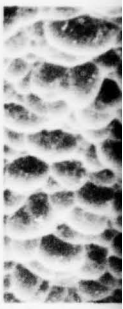
Groun



Ground with 3 μm Al_2O_3, and 0.5-hour HF etch



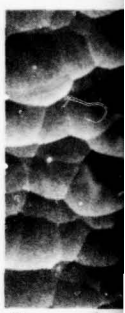
Ground with 9 μm Al_2O_3 and 0.5-hour HF etch



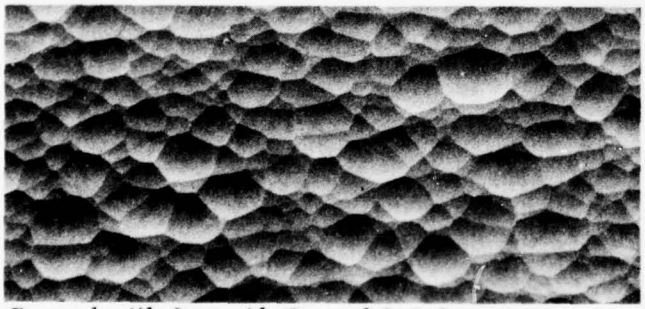
Ground w

Ground with 3 μm Al_2O_3 and 1.0-hour HF etch

Ground with 9 μm Al_2O_3 and 1.0-hour HF etch



Ground w



Ground with 3 μm Al_2O_3 and 2.0-hour HF etch

Ground with 9 μm Al_2O_3 and 2.0-hour HF etch

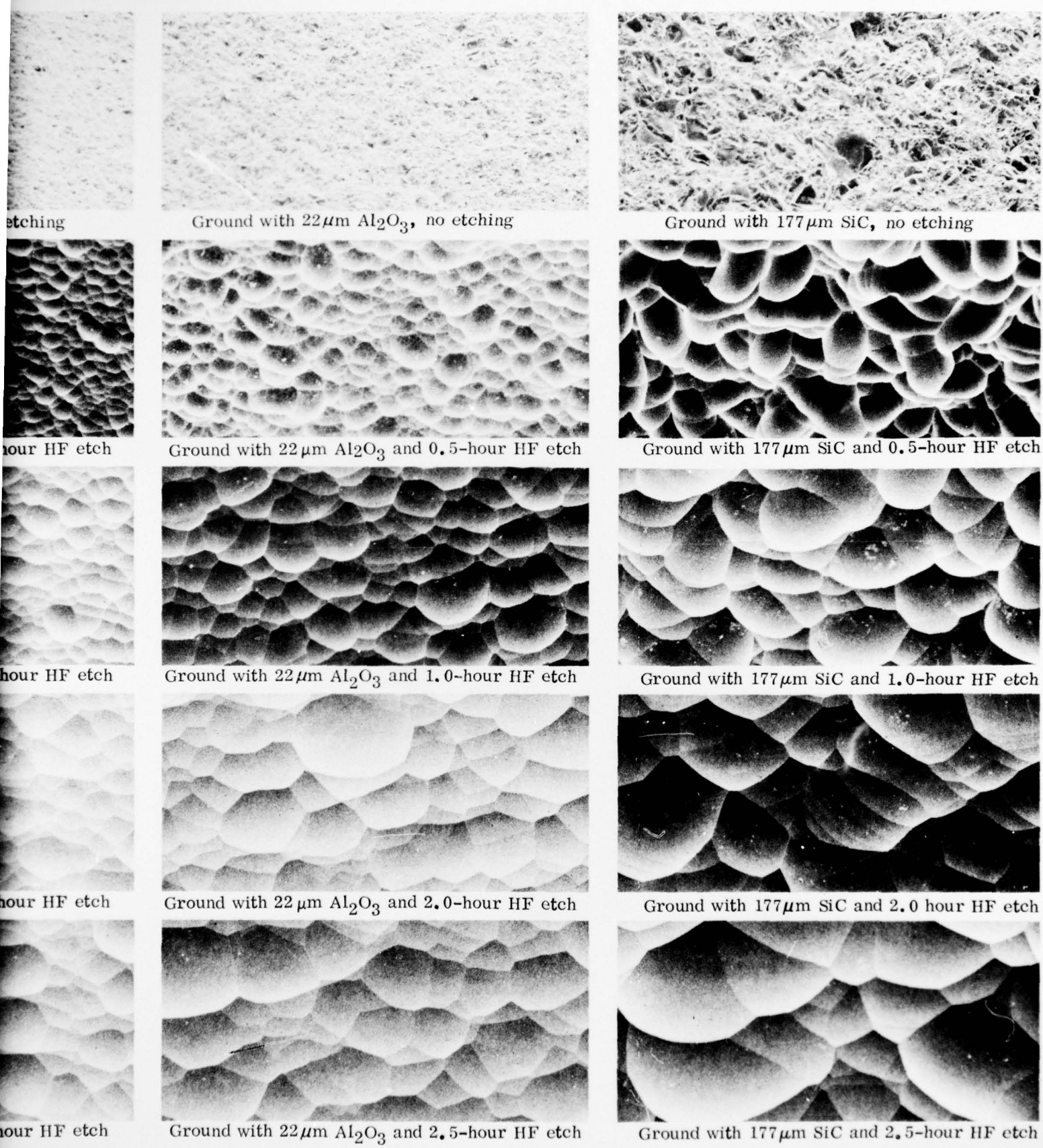
Ground w

Ground with 3 m Al_2O_3 and 2.5-hour HF etch

Ground with 9 μm Al_2O_3 and 2.5-hour HF etch

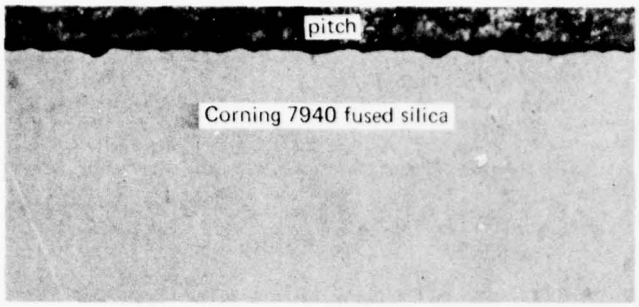
Ground

Figure 7-1



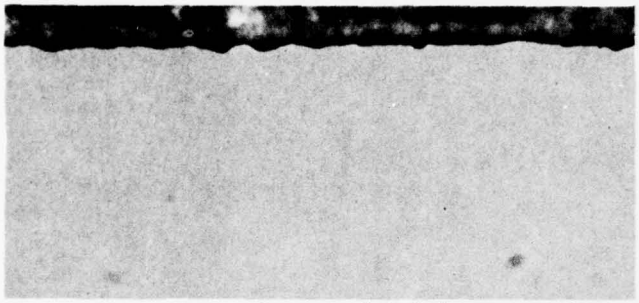
Corning 7940 fused-silica – scanning electron microscope micrographs. (300X magnification in all cases).

Figure 7-1

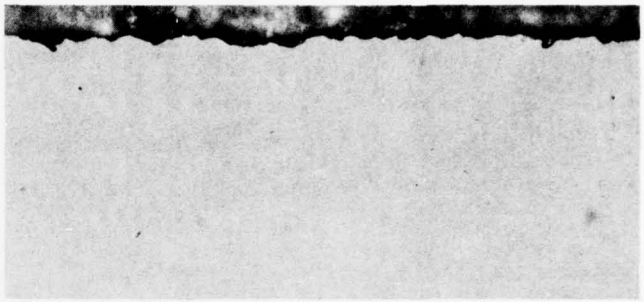


Ground with 3μm Al_2O_3, no etching

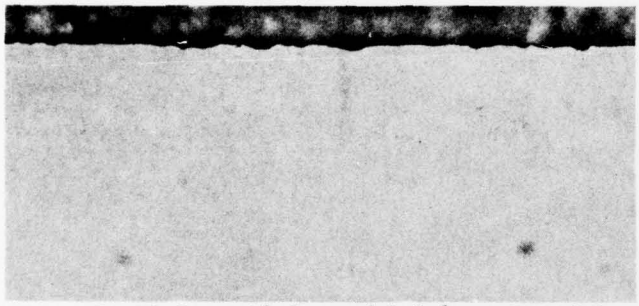
Ground with 9μm Al_2O_3, no etching



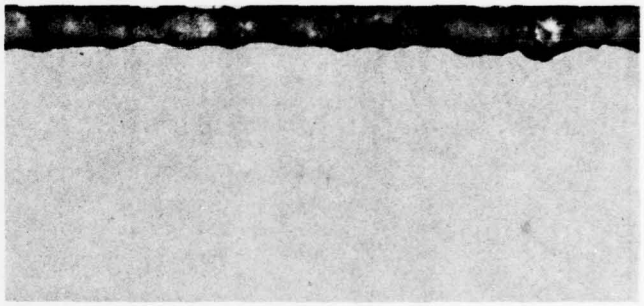
Ground with 3μm Al_2O_3 and 0.5-hour HF etch



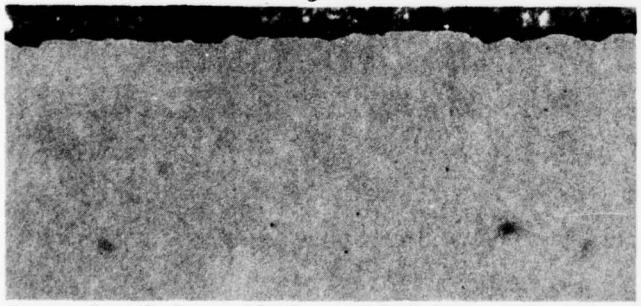
Ground with 9μm Al_2O_3 and 0.5-hour HF etch



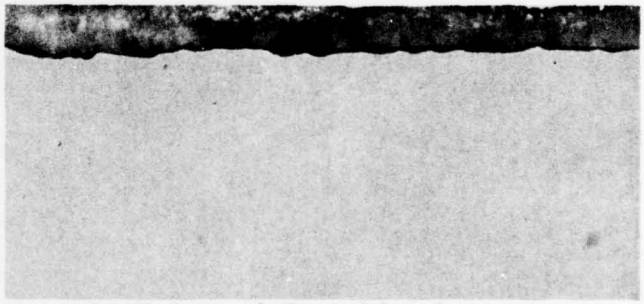
Ground with 3μm Al_2O_3 and 1.0-hour HF etch



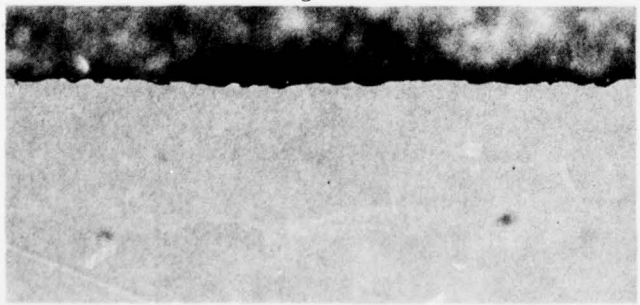
Ground with 9μm Al_2O_3 and 1.0-hour HF etch



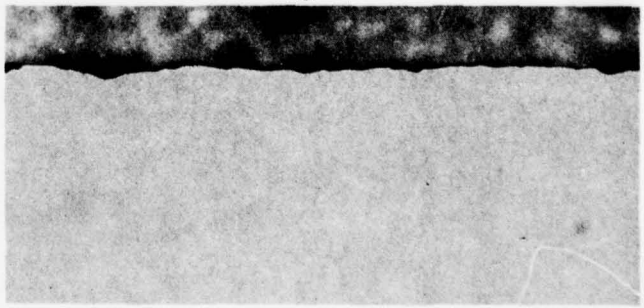
Ground with 3μm Al_2O_3 and 2.0-hour HF etch



Ground with 9μm Al_2O_3 and 2.0-hour HF etch

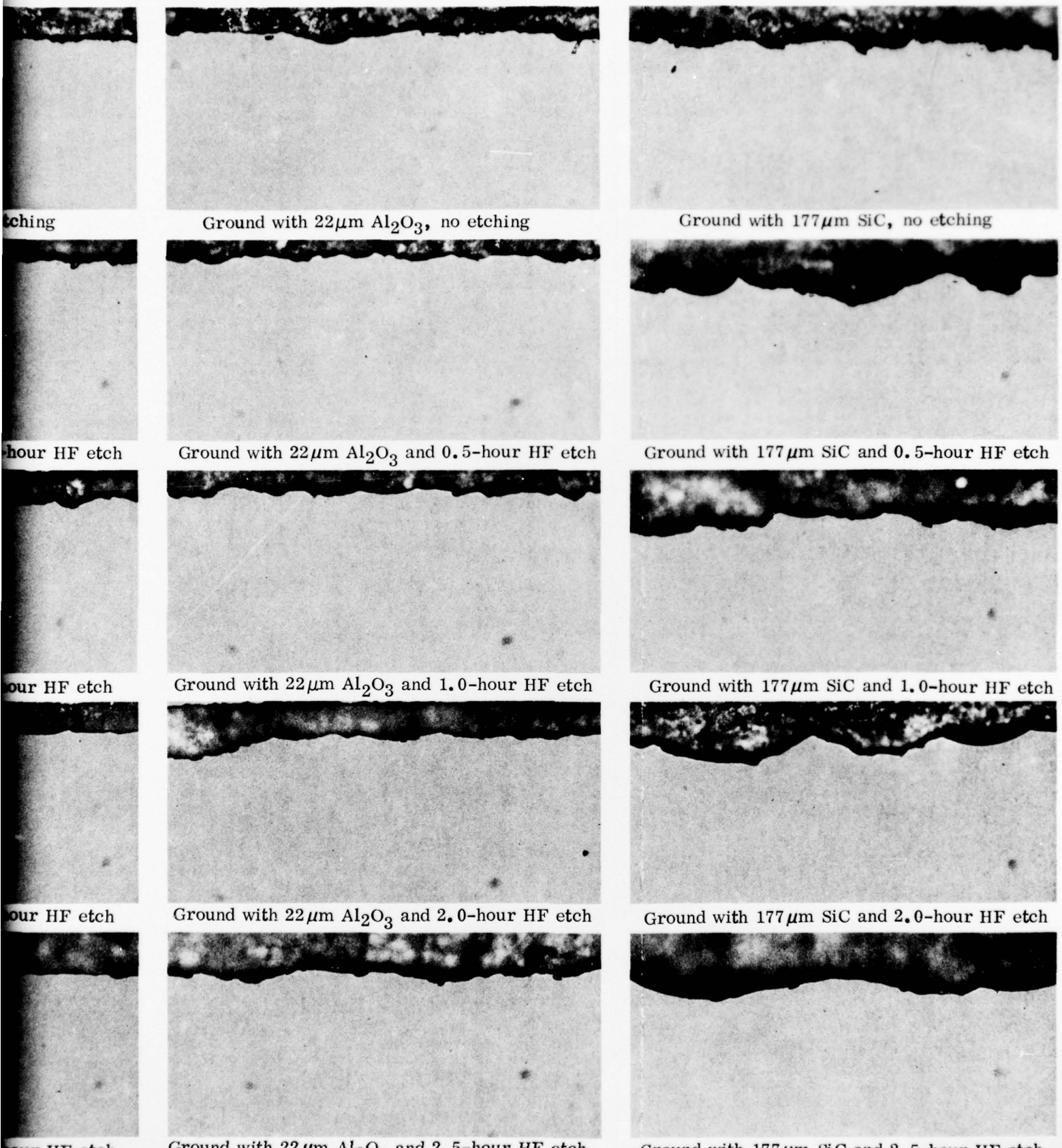


Ground with 3μm Al_2O_3 and 2.5-hour HF etch



Ground with 9μm Al_2O_3 and 2.5-hour HF etch

Figure 7-2



Corning 7940 fused silica – profile photographs with optical microscope (300X magnification in all cases)

Figure 7-2

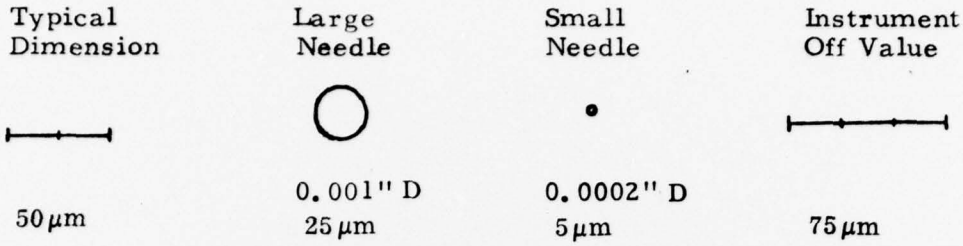


Figure 7-3. Scaled-Up Features of Profilometer Scanning Scaled Up 300 Times

This needle was later replaced by a smaller one of 0.0001 in.R, which is also represented in Figure 7-3, at the scale of the photographs (300X). This needle could serve to trace the profile, except for the fact that the electronic equipment has a limiting cut-off of 0.003 in. (75 μm on the photographs — see Figure 7-3) for the vibrations that can be detected and analyzed. This limitation is related to the mechanical compliance of the arm, external vibrations and other sources of noise both of vibrational and electronic origin.

The net result was that the equipment "skimmed" over the surface and failed to produce a true profile. This type of measurement was destructive since the needle, because of the pressure in the arm, knocked off the sharp edges leaving a sort of channel in the surface clearly detectable in the microscope at 300X magnification.

The method finally used to determine the profile involved cladding a sample ground and etched fused silica wafer between two smooth sheets of fused silica, as shown in Figure 7-4. The edges were ground and polished and then photographed with a light microscope. Because the pitch was softer than the silica, and because of the heat generated during the grinding and polishing process, the fused silica tended to crack and chip along the edges to be studied. The polishing operation was repeated several times before well-cut, sharp edges could be obtained. Eventually satisfactory polishes were obtained and the resulting photographs are shown in Figure 7-2. The arrangement of pictures is the same as in Figure 7-1.

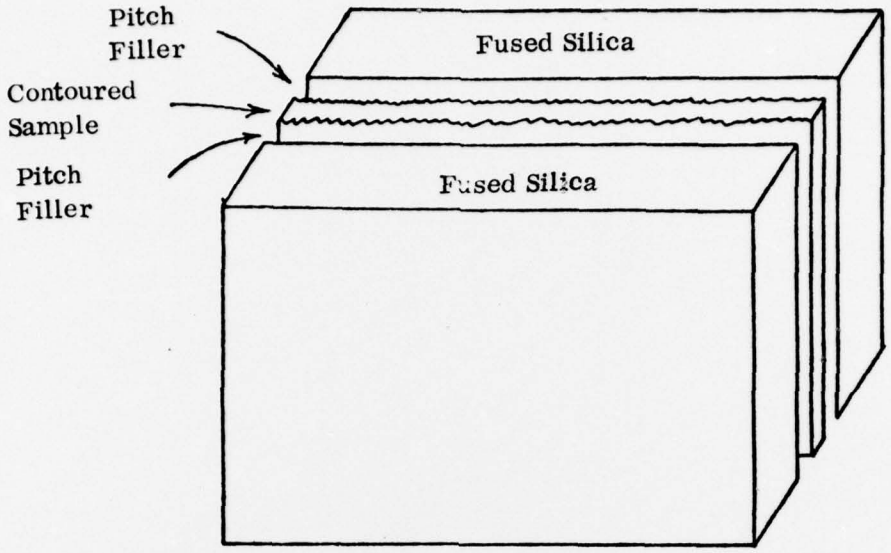


Figure 7-4. Physical Arrangement for Obtaining Cross-Section Photographs

SECTION 8

SURFACE OPTICAL PROPERTIES

The bidirectional reflectance at $\lambda = 0.5\,\mu m$ and directional-hemispherical reflectance (UV, VIS, IR) of the eight samples selected for full-scale testing were determined. From the directional-hemispherical reflectance, the thermal emittance and solar reflectances were derived. The instrumentation, experimental procedures, and data compilation methods are presented in Annexes I, II and II of Volume II. The data are summarized in Table 8-1. In Annexes V, VI, VIII, and IX of Volume II the data are given in some detail.

Table 8-1. Summary of Optical Property Data

Mirror Type (1)	ρ_s	ϵ_n	ϵ_h	$\rho'_{20°}$	$\rho'_{60°}$	α_s/ϵ_h
FS 3 μm 9 μm 1.5 N	.928	.845	.790	1.42	4.7	.091
FS 3 μm 9 μm 2.5 N	.931	.846	.791	2.3	5.8	.087
FS 30 μm-P .5 N	.919	.873	.816	2.1	2.9	.099
FS 3 μm 9 μm 1.5 E	.927	.831	.777	1.5	4.5	.093
FS 3 μm 9 μm 2.5 E	.942	.829	.775	2.2	6.6	.075
FS 30 μm P .5 E	.918	.863	.806	2.1	2.4	.10
FEP 3 μm 9 μm Q .85	.893	.874	.817	2.0	4.5	.13
FEP 3 μm 9 μm Q .85	.900	.889	.831	2.0	4.6	.12
DESIGN GOALS	.94		.77	2.0		.078
OCLI SI-100(Non En)	.945					
OCLI MSI-100 (En)	.972					
GD/CV Std (Non En)	.947					

1) See Section 5.1 for description.

2) ρ_s = Solar Reflectance, 18° angle of incidence

3) ϵ_n = Near Normal Emittance

4) ϵ_h = Hemispherical Emittance (obtained by applying factor of 0.935 to ϵ_n ref. Eckert and Drake)[3]

5) $\rho'_{20°}$ = Maximum Bidirectional Reflectance for Polar Incidence Angle of 20^o

6) $\rho'_{60°}$ = Maximum Bidirectional Reflectance for Polar Incidence Angle of 60^o

7) α_s/ϵ_h = Solar Absorptance/Hemispherical Emittance

8.1 SOLAR REFLECTANCE

The first column in Table 8-1 gives the solar reflectances. The third digit in the values is not justifiable on the basis of absolute accuracy. It is included since it probably has some significance for comparison between samples. Also included in the table are determinations on OCLI regular silver (SI-100), enhanced silver (MSI-100) and a Convair regular silver standard. These three samples were all specular.

It is seen that the OCLI regular silver and the Convair non-enhanced specular standard show solar reflectances of 0.945 and 0.947 respectively. The specification for the OCLI sample is 0.94 or greater.

The integrating sphere Cary traces of the Convair standard and the OCLI SI 100 are identical except from about $\lambda = 0.33$ to $0.44\,\mu m$, in which interval the Convair standard is a better reflector by 2 to 3%. This influences the solar reflectance by only a small amount, 0.945 versus 0.947.

The measured bidirectional reflectance in units of sr^{-1} at the specular angle for incidence angles of 20 to 60 deg. are given in Table 8-1 in the columns under $\rho'_{20°}$ and $\rho'_{60°}$. It was desired to make the mirrors as close as possible to diffuse reflectors. A perfectly diffuse reflector would have a BRDF of $\frac{1}{\pi}$ sr^{-1} at all angles. A perfect specular mirror would have a bidirectional reflectance of infinity at the specular angle, zero at all other angles. Current commercial second surface mirrors have a bidirection reflectance in excess of 3000 sr^{-1} at the specular angle, zero at other angles. The design goal in this program was 2 sr^{-1}. The incidence angle for this value was not designated. It is seen that all samples are in the 2 sr^{-1} range at 20° incidence, but at 60° incidence the values are higher. The 30 μm-polished samples are less than 3 sr^{-1} at 60°. It will be seen in the detailed data that the superior scattering performance of the 30 μm roughened samples is always evident at large angles and is a plus factor for this design.

The near-normal directional emittance is given in the column under ϵ_n and the value for hemispherical ϵ_h emittance is derived from this value by multiplication by a factor 0.935 as obtained from Eckert and Drake. (3)

Regarding the hemispherical emittance, ϵ_h, it is seen that for the fused silica samples, the 3 μm-9 μm designs have a slightly lower emittance than the 30 μm-polished samples, irrespective of the type of silver. The greater roughness of the 30 μm-polished samples probably accounts for this difference, a rougher surface having a higher emittance. The effect on α_s/ϵ_h is disappointingly trivial.

The last column gives the solar absorptance divided by the hemispherical emittance α_s/ϵ_h a ratio which describes the net performance of a thermal control coating. Sample FS 3 μm-9 μm 2.5 E meets design goals.

Optical property data are provided in the annexes of Volume II as follows:

Annex V

Tabulated reflectance at closely spaced wave lengths from 0.3 to 7 μm and computed solar absorptance. Solar data used: air mass zero spectral irradiance, Reference ASTM E 490-73.[4]

Annex VI

Tabulated reflectance from 0.3 to 29 μm in ERAS format. Solar absorptance. Solar data used: air mass zero spectral irradiance, Reference ASTM E 490-73.[4]

Annex VII

Tabulated reflectance from 2.5 to 30 μm and computed total hemispherical emittances for 200K, 250K, 300K, 350K, 400K, 450K, 500K, 550K, 600K, 650K and 700K.

Annex VIII

Plotted directional-hemispherical reflectance 0.3 to 30 μm.

Annex IX

Two-dimensional and three-dimensional representations of bidirectional reflectance data.

SECTION 9

PHYSICAL PROPERTY TESTING

Military Specification testing was performed as called out in paragraph 3.0 (Military Specification Tests) of the Annex IV of Volume II.

9.1 APPEARANCE

The coatings observed by the unaided eye gave the appearance of uniform coverage. The uncoated surface was free of all metal deposition and other contaminations. The overcoated back surface had a distinct color when viewed in white light.

9.2 COATING ADHERENCE

Humidity resistance, coating hardness, thermal cycling and a recheck of coating adherence. These tests were all performed as called out in the Annex IV of Volume II and with the equipment therein described. Figure 9-1 shows the samples in the humidity chamber. The results are presented in Table 9-1.

9.3 *DISCUSSION OF PHYSICAL PROPERTY TEST RESULTS*

All fused silica samples passed the hardness, tape tests, boiling water, and tape test after boiling water. The enhanced samples failed the humidity-thermal cycling tests. The damage apparently propagated from the edge. Sealing the edges of the sample against water might well cure this difficulty.

The nonenhanced samples passed the tape test, hardness test, boiling water test, and tape test after boiling water. Samples presenting a specular surface to the coating (FS 30-P.5 N-4 and Standard N-4) passed the humidity and thermal cycling tests with no damage. Very small pin holes appeared on the samples presenting an etched surface to the coating when subjected to humidity and thermal cycling. The damage was not extensive, less than 1%. The difficulty may be due to improper cleaning and alternate cleaning methods may be required for etched surfaces which are to be coated. The cleaning methods used are clearly suitable for the specular surface.

The FEP Teflon passed the tape test and hardness test, but failed otherwise. A control on a plain sample was not included.

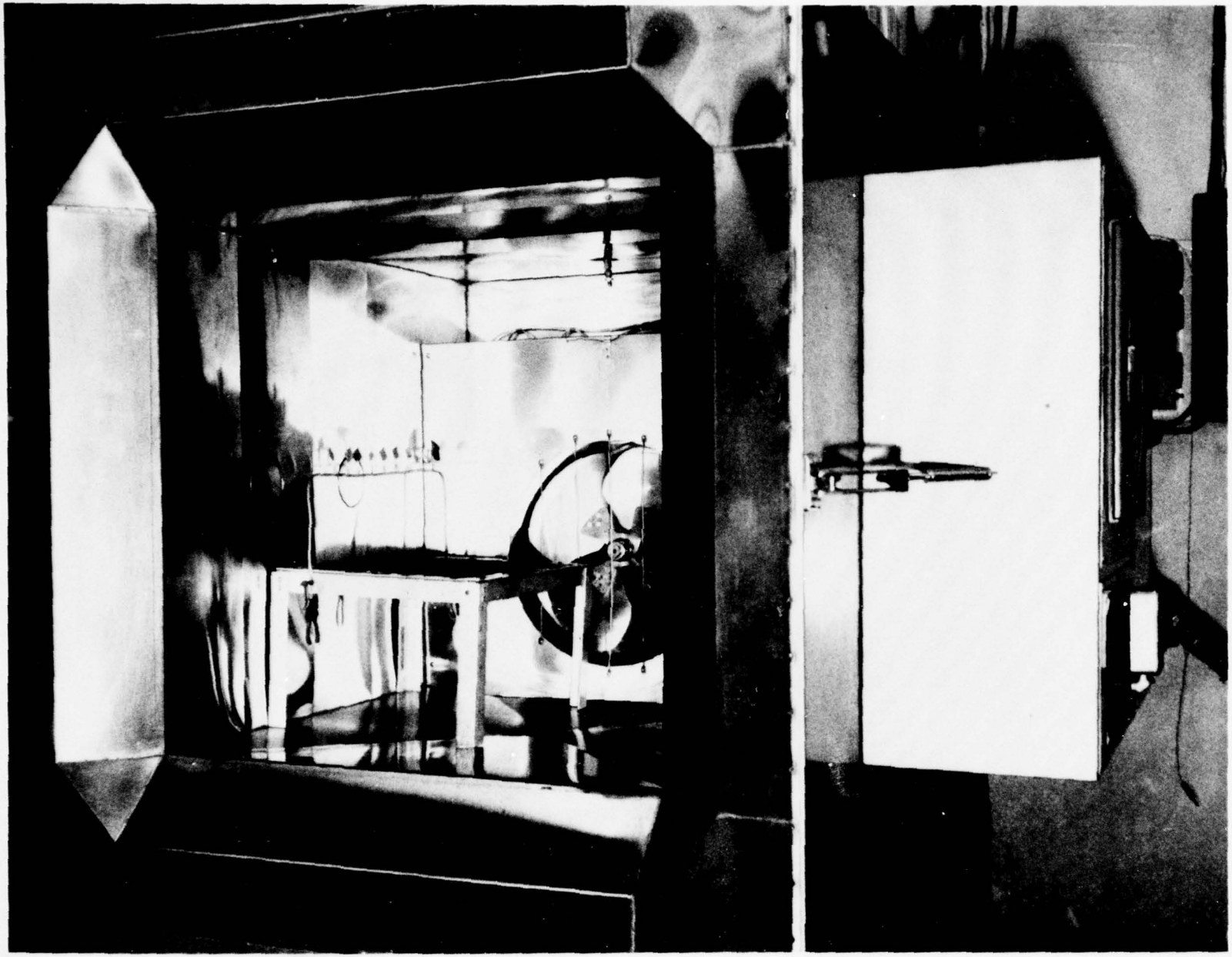


Figure 9-1. Humidity Chamber.

Table 9-1. Physical Property Testing Results

SAMPLE	TAPE TEST	BOILING WATER	TAPE TEST	HARDNESS	HUMIDITY and THERMAL CYCLING(1)	TAPE TEST	BOILING WATER	TAPE TEST
FS 3μm- 9μm1.5 E-6	X	X	X	X	F(2)			
FS 3μm- 9μm2.5 E-6	X	X	X	X	F(2)			
FS 30μm-P .5 E-6	X	X	X	X	F(2)			
FS 3μm- 9μm1.5 N-4	X	X	X	X	X(3)	X(3)		
FS 3μm- 9μm2.5 N-4	X	X	X	X	X(3)	X(3)		
FS30μm- P .5 N-4	X	X	X	X	X	X		
Standard N-4	X	X	X	X	X	X		
FEP 3μm- 9μmQ.83-1	X	F						
FEP 3μm- 9μmQ.83-2	X	F						
FEP 3μm- 9μmQ.83-3			X	X	X	F		
FEP 3μm- 9μmQ.83-4			X	X	X	F		

X=Passed

F=Failed

(1) Humidity and thermal cycling tests were performed sequentially and samples were inspected after both tests were complete.

(2) Damage seemed to have propagated from edge of water.

(3) Pin point hole damage.

SECTION 10

REFERENCES

1. S. J. Schneider, Jr. and R. W. Rice, "The Science of Ceramic Machining and Surface Finishing," NBS Publication 34.8, May, 1972.

2. Frekote 33 is the product of Frekote, Inc., Boca Raton, FA.

3. E. R. G. Eckert and R. M. Drake, "Heat and Mass Transfer," McGraw-Hill Book Co., 1959, page 380.

4. E490-73a, "Solar Constant and Air Mass Zero Solar Spectral Irradiance, Spec. for," Part 41, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.

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